GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. _____

Project No. A-60-602 SUB IS G-35-805 GTRI/~~GTK~~ DATE 6 / 8 / 84

Project Director: Dr. R. A. Karam School/~~Lab~~ Nuclear Reactor Center

Sponsor: E. I. DuPont de Nemours & Co.; Aiken, SC 29808-001

Type Agreement: Purchase Order No. AX-0654690 under DOE Prime

Award Period: From 5/11/84 To 3/30/85 (Performance) 3/31/85 (Reports)

Sponsor Amount:	This Change	Total to Date
Estimated: $		$ 56,483
Funded: $		$ 56,483

Cost Sharing Amount: $ _____ Cost Sharing No: _____

Title: Characterizing Changes in Adsorbents Properties Under High Radiation Doses

ADMINISTRATIVE DATA — OCA Contact John W. Burdette x4820

1) Sponsor Technical Contact:

F. C. Lucius
E. I. DuPont de Nemours & Co.
Savannah River Plant
Aiken, SC 29808-0001

(803) 725-2695

2) Sponsor Admin/Contractual Matters:

Kurt Friedrickson
E. I. DuPont de Nemours & Co.
Savannah River Plant
Aiken, SC 29808-0001

(803) 725-3981

Defense Priority Rating: n/a

Military Security Classification: n/a

(or) Company/Industrial Proprietary: n/a

RESTRICTIONS

See Attached ------ Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval – Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with Gov't. with an acquisition cost of $1,000 or more; vests with GT having an acquisition cost of less that $1,000 if prior approval is obtained from DuPont.

COMMENTS:

Sponsor I.D. #02.204.000.84.001

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FORM OCA 4:383

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date January 7, 1986

Project No. A-60-602 School/~~Unit~~ NRC

Includes Subproject No.(s) G-35-805

Project Director(s) Dr. R.A. Karam GTRC / ~~GTI~~

Sponsor E.I. DuPont De Nemours and Company

Title Characterizing Changes in Adsorbents Properties Under High Radiation Doses

Effective Completion Date: 6/30/85 (Performance) _____ (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [x] Final Invoice or Final Fiscal Report
- [x] Closing Documents
- [x] Final Report of Inventions
- [x] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other _____

Continues Project No. _____ Continued by Project No. _____

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FORM OCA 69.285

A-60-602

Georgia Institute of Technology

A UNIT OF THE UNIVERSITY SYSTEM OF GEORGIA

SCHOOL OF MECHANICAL ENGINEERING

July 12, 1984

Please reply to:

NUCLEAR ENGINEERING AND
HEALTH PHYSICS PROGRAM
CHERRY EMERSON BUILDING
GEORGIA INST. OF TECH.
ATLANTA, GEORGIA 30332 U.S.A.

Dr. Paul R. Monson
Actinide Technology Div.
E. I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This letter is a Progress report for the month of June 1984 on the project entitled "Characterization of Changes in Absorbent Properties under High Radiation Doses".

1. Sample Holders

The number of sample holders purchased and assembled are 48. Table 1 lists the experimental conditions for all 48 holders. The first four "control" sample holders should measure what happens to the air inside the stainless steel holders which contain no zeolite, but in one case exposed to 10^{10} rads and in other not irradiated at all. Mass spectrometry will be used to analyze the air. The air in the unirradiated case will have resided in the holder the same length of time as in the irradiated case.

The next four sample-holders monitor pressure and temperature on a daily basis, during the entire exposure period.

The next 8 samples will not be irradiated but one silver- and one hydrogen-zeolite sample will be analyzed along with irradiated samples at each appropriate dose.

Figure 1 shows all 48 sample holders assembled.

2. Gas Mixing

One dry air bottle containing 0.5% He and one small NO_2 bottle were ordered and received from Matheson. Ozone will be generated at Georgia Tech Environmental Research Laboratory. A 10-liter glass bottle with the appropriate valving has been obtained through the School of Chemistry Glass Blowing Shop. The plan is to evacuate the glass bottle and then introduce the NO_2 to the right partial pressure (mmH_2O), add O_3 to the appropriate partial pressure (7mm H_2O) and add air with 0.5%. He up to a total pressure of 1166 mm of Hg. The Ozone from the Ozone generator will be analyzed by the wet chemical method and mass spactrometry for ozone content. The mixture of gases in the 10-liter bottle will be analyzed by mass spectrometry. The 10-liter jar is shown in Figure 2 along with the Sample holders.

3. Adsorption measurements and X-Ray Diffraction Calibration

The absorption measurements will be carried out using McBain-Bakr gravimetric system, which employs a spiral quartz spring on which the sample is suspended in a pan. The extension of this spring is a direct measurement of the weight of the gas absorbed. Attached are the calibration curves (weight versus extension) for the six quartz springs that will be used in this work.

The X-ray diffraction equipment is calibrated each day with an internal standard.

4. Zeolite Crushing and Hardness Tests

ASTM Standard D-4179-82 (Appended) was used for this test. Fifty samples of each of the hydrogen, and Silver-Zeolites were used in accordance with the standard. The results are given in Table II. In both cases the standard deviation is large. Both, the hydrogen-and Silver-Zeolites are rather weak, especially the Silver. It is not known if the heating of the Zeolite samples, as called for by the ASTM Standard for the crushing test, is contributing to the weakening of the pellets. It was found that after heating, standard hardness tests can not be applied because pellets were too weak. New tests are being devised for the hardness test.

I have asked Dr. James Benzel of Ceramic Engineering to measure the hardness and crush resistance of the pellets without heating the samples. As soon as these results are available I will communicate them to you.

This is basically where we are on this project. I anticipate irradiation will begin soon. If you have any questions please let me know.

Best wishes

R. A. Karam
Director

RAK/swm

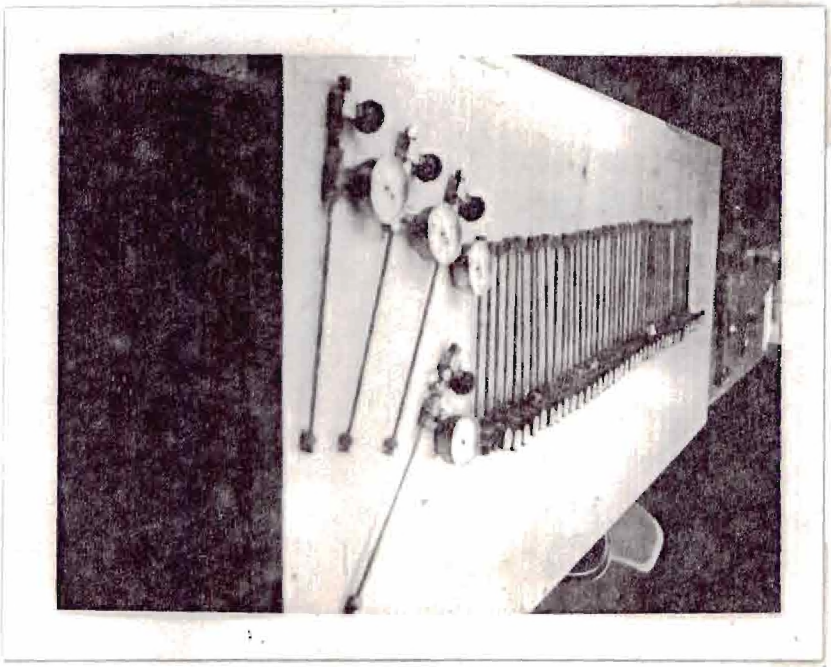


Figure 1. Sample Holders for Hydrogen- and Silver-Zeolite Irradiation

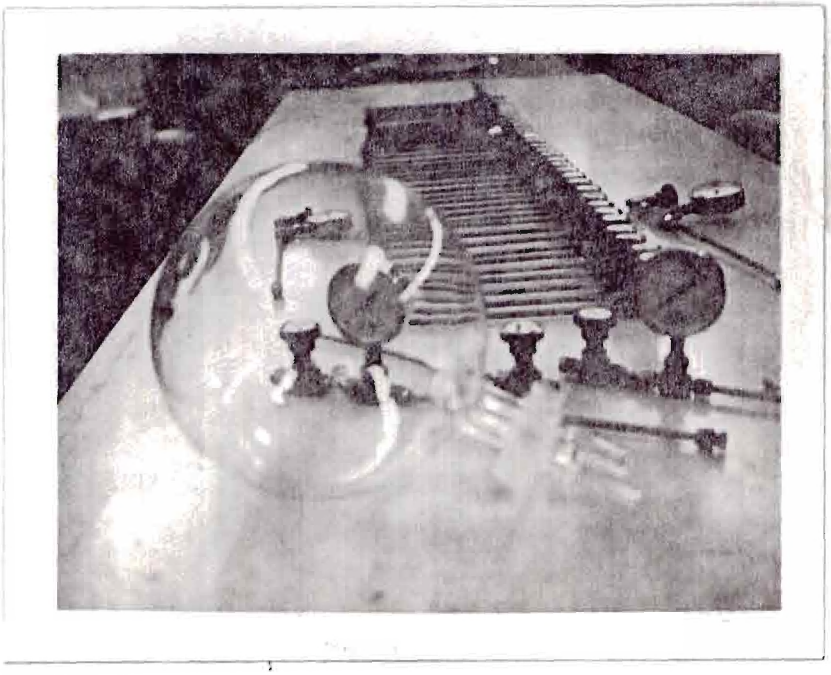


Figure 2. 10-Liter Glass Bottle and Sample Holders.

TABLE 1. SAMPLE HOLDERS AND EXPERIMENTAL CONDITIONS

TYPE OF SAMPLE	QANTY	TOTAL DOSE, RADS	EXPERIMENTAL CONDITIONS
Control	1	$1x10^{10}$	No Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	not irradiated*	No Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	$1x10^{10}$	No Zeolite in sample holder, Saturated air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	not irradiated*	No Zeolite in sample holder, Saturated air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	10^{10}	Silver Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	10^{10}	Hydrogen Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Control	1	10^{10}	Silver Zeolite in sample holder, Sat. air (1000 PPMO_3; 1000 PPM No_2)
Control	1	10^{10}	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	not irradiated*	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM No_2)
Samples	2	not irradiated*	Hydrogen Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	not irradiated*	Silver Zeolite in sample holder, Dry air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	not irradiated*	Silver Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	$1x10^{8}$	Silver Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	$1x10^{8}$	Silver Zeolite in sample holder, Dry air (1000 PPM)$_3$; 1000 PPM NO_2)
Samples	2	$1x10^{8}$	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM NO_2)
Samples	2	$1x10^{8}$	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM 0_3; 1000 PPM NO_2)

Samples	2	$1x10^9$	Hydrogen Zeolite in sample holder, Sat. air (1000 O_3; 1000 PPM No_2)
Samples		$1x10^9$	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$1x10^9$	Silver Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$1x10^9$	Silver Zeolite in sample holder, Dry air (1000 PPMO_3; 1000 PPM No_2)
Samples	2	$5x10^9$	Silver Zeolite in sample holder, Dry air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$5x10^9$	Silver Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM No_2)
Samples	2	$5x10^9$	Hydrogen Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$5x10^9$	Hydrogen Zeolite in sample holder, Dry air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$1x10^{10}$	Hydrogen Zeolite in sample holder, Dry air (1000 PPM O_3; 1000 PPM No_2)
Samples	2	$1x10^{10}$	Hydrogen Zeolite in sample holder, Sat air (1000 PPM O_3; 1000 PPM NO_2)
Samples	2	$1x10^{10}$	Silver Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM No_2)
Samples	2	$1x10^{10}$	Silver Zeolite in sample holder, Sat. air (1000 PPM O_3; 1000 PPM NO_2)

*Analyzed at same time as irradiated samples

TUBE 130 ΔH vs w

ΔH = H_oof − H_with weight

H_oof = height with empty bucket

y = mx + b

hope fully b = 0

hence y = mx

ΔH	w (g)
5.085	.0860
9.475	.1619
13.550	.2099
20.105	.3402
26.185	.4429
29.525	.4996
35.010	.5931
37.190	.6301

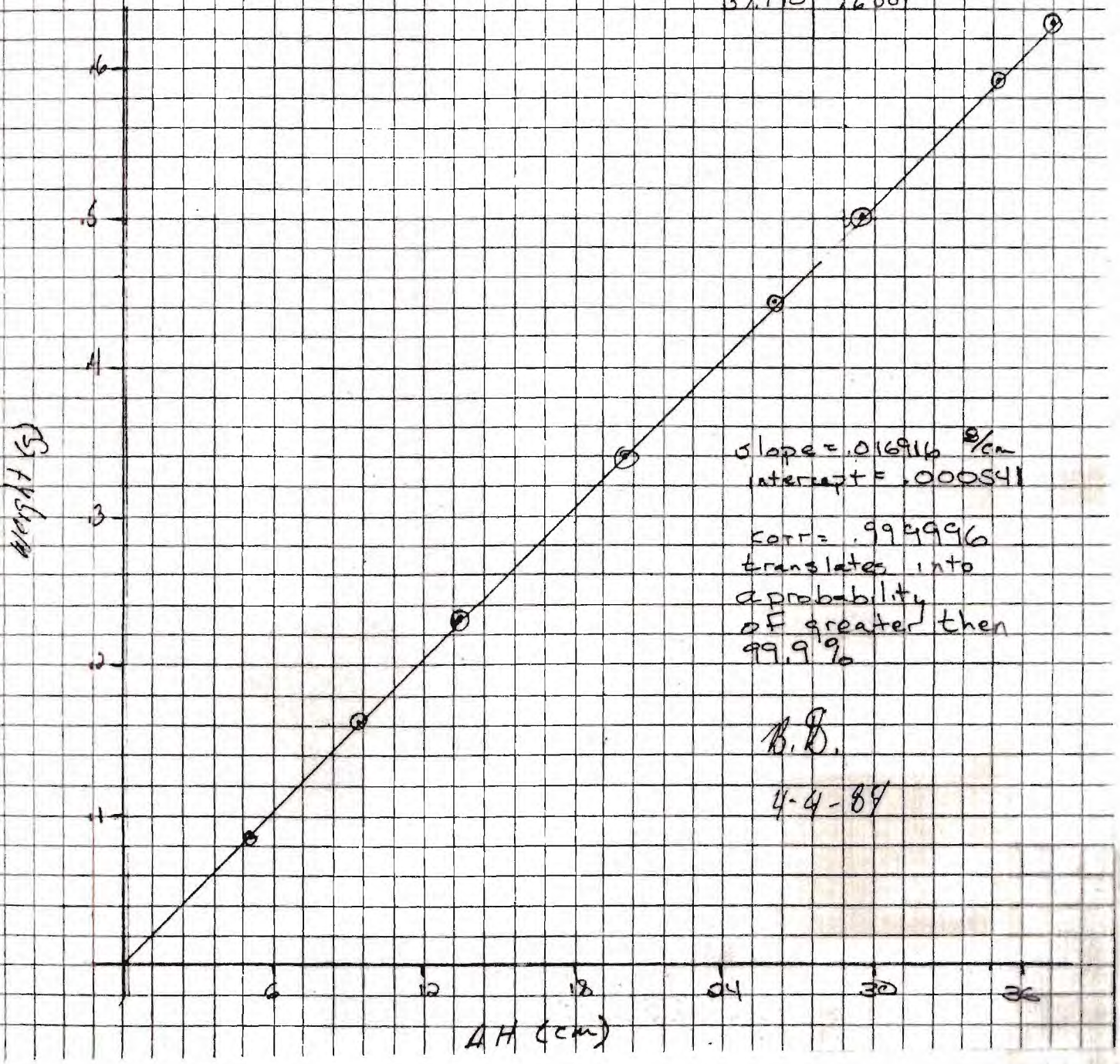


Fig. 3 Weight of Gas Adsorbed Versus Spring displacement for Tube 130

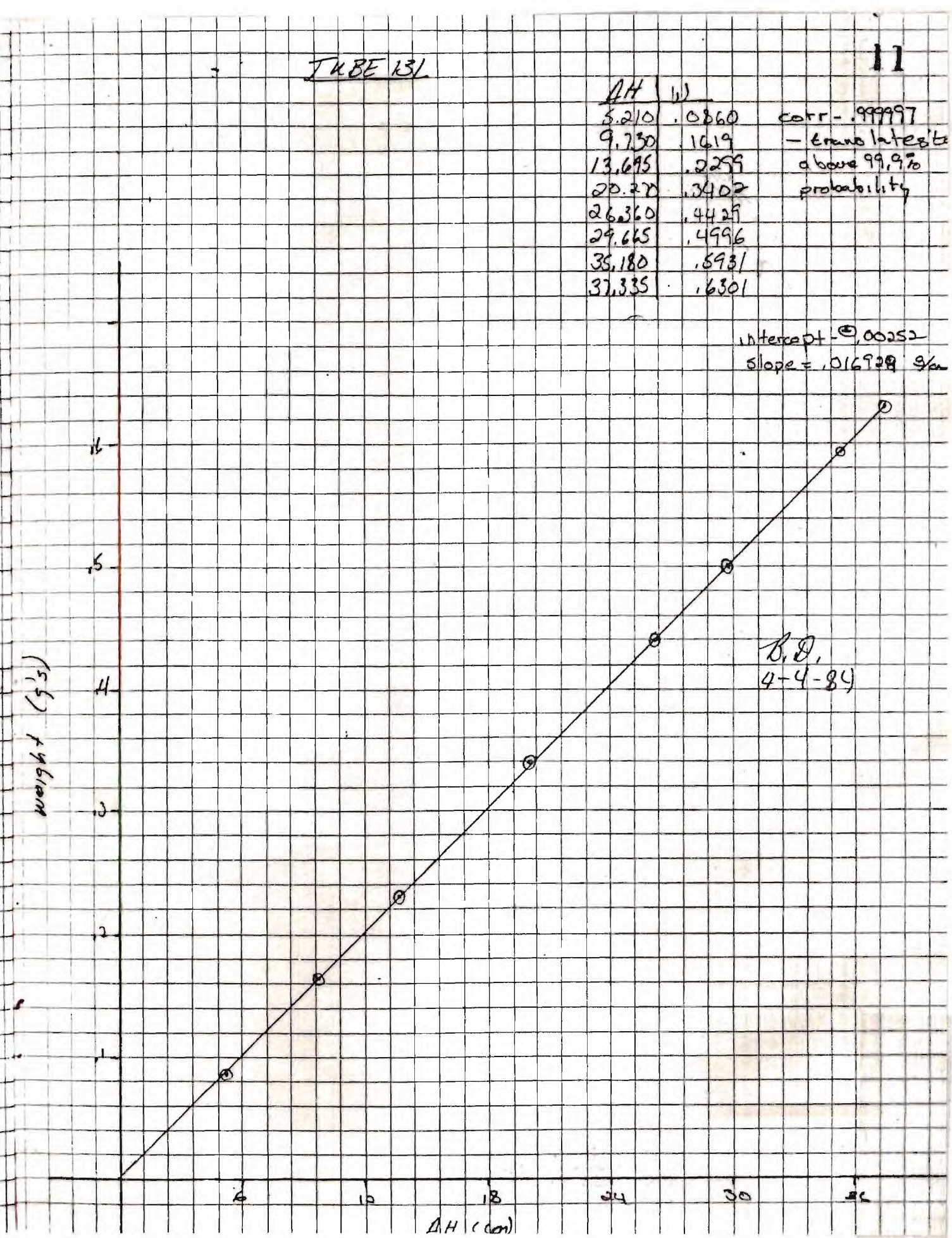


Fig. 4 Weight of Gas Adsorbed Versus Spring Displacement for Tube 131

TUBE 132

ΔH	w
5.240	.0860
9.765	.1619
13.840	.2299
20.325	.3402
26.475	.4429
29.820	.4996
35.325	.5931
37.455	.6301

corr - .9999945 - above a 99.9% probability
intercept + 0.002927
slope - .0168634 g/cm

B.B
4-4-84

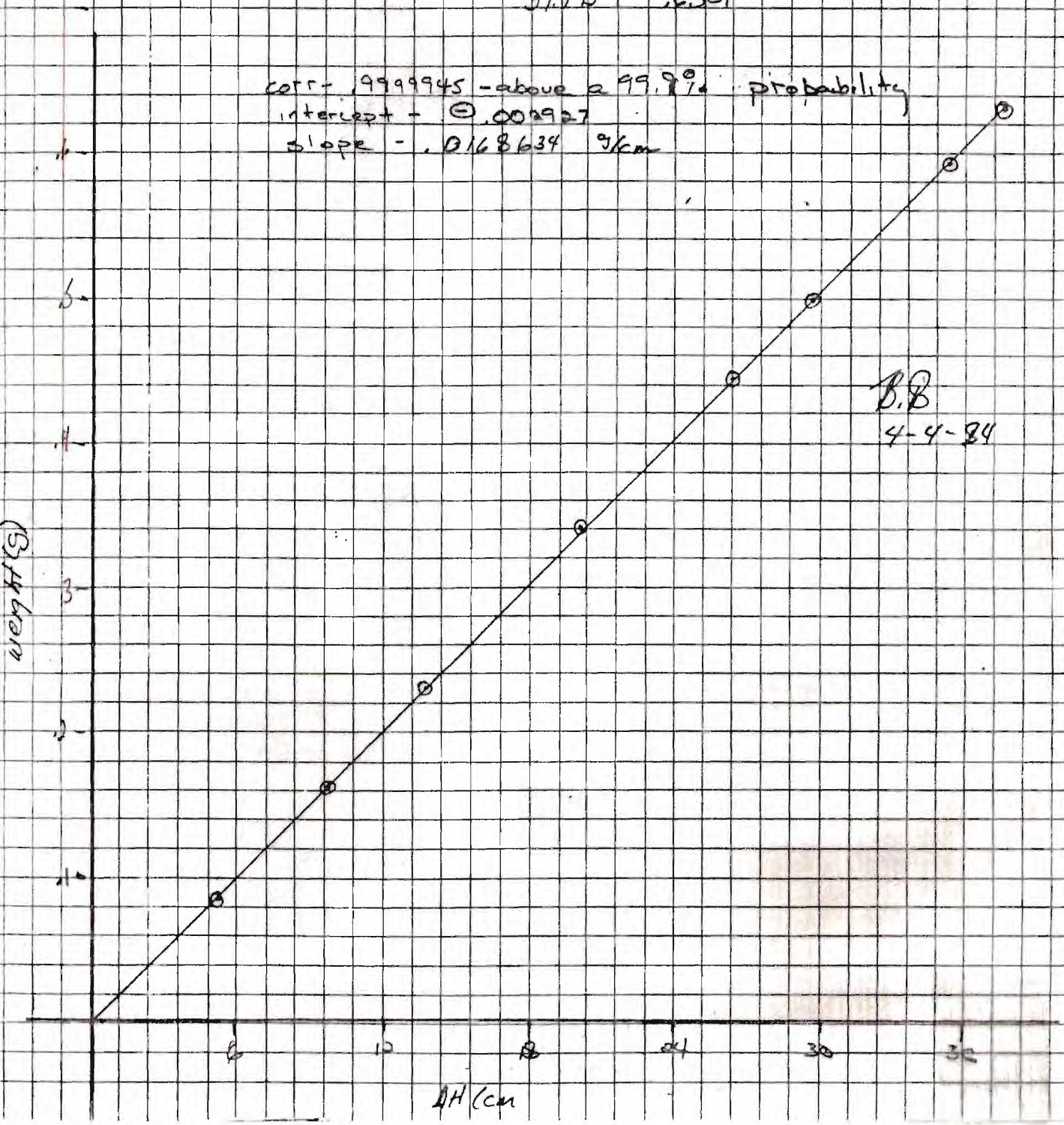
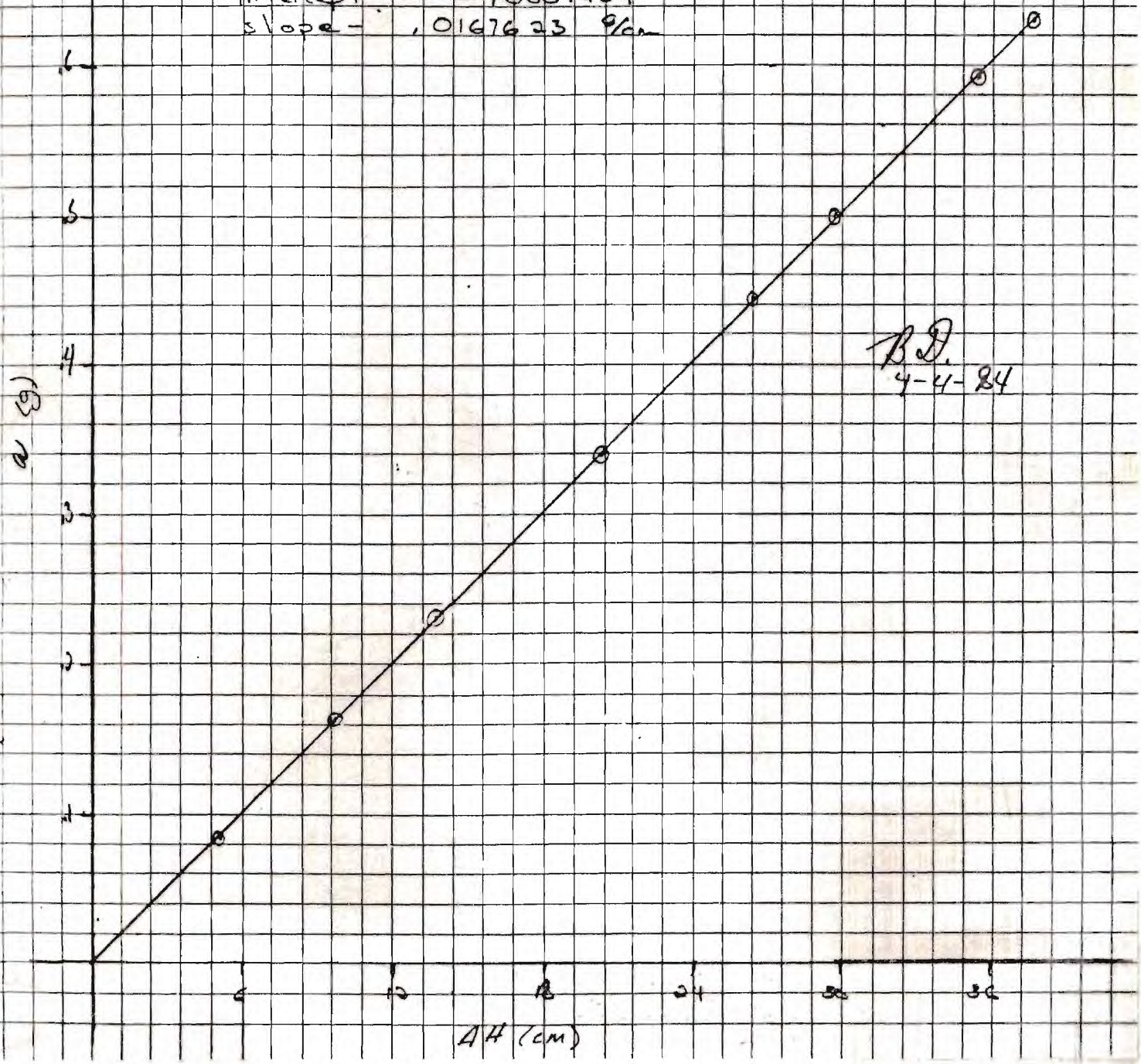


Fig. 5 Weight of Gas Adsorbed Versus Spring Displacement for Tube 132

TUBE 133

ΔH	W
5.125	.0860
9.645	.1619
13.705	.2299
20.355	.3402
26.445	.4439
29.825	.4960
35.365	.5931
37.585	.6301

corr - .9999978 - probability above 99.9%
intercept - 0.0001454
slope - .0167623 g/cm



B.D.
4-4-84

Fig. 6 Weight of Gas Adsorbed Versus Spring Displacement for tube 133

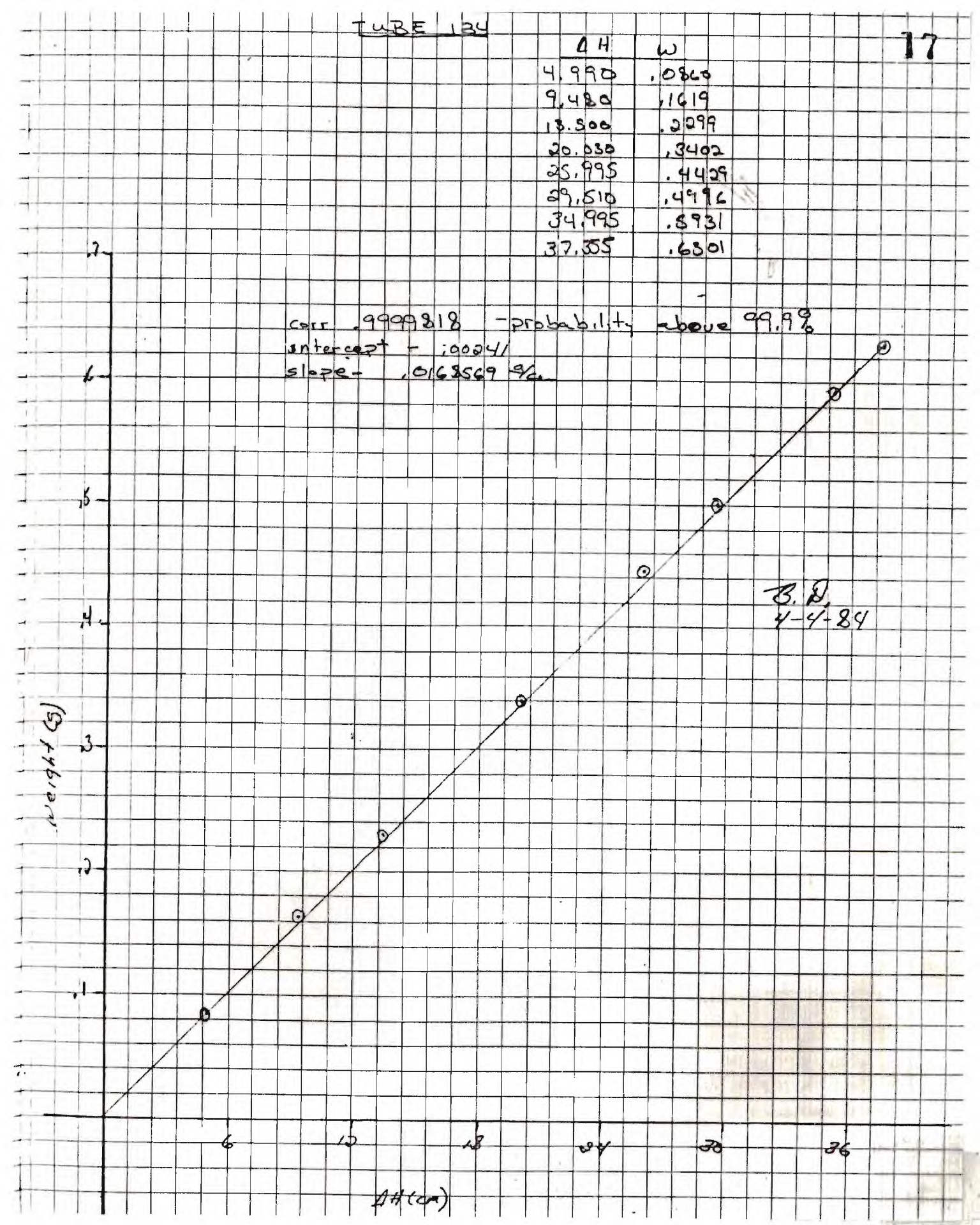


Fig. 7 Weight of Gas Adsorbed vs. Spring Displacement for tube 134

TUBE 135

ΔH	W
5.105	.0860
9.615	.1619
13.645	.2299
20.185	.3402
26.270	.4429
29.655	.4996
35.190	.5931
37.315	.6301

Corr .9999985 probability above 99.9%
Intercept - 0.000483
slope - .0168856 g/cm

R D
4-4-84

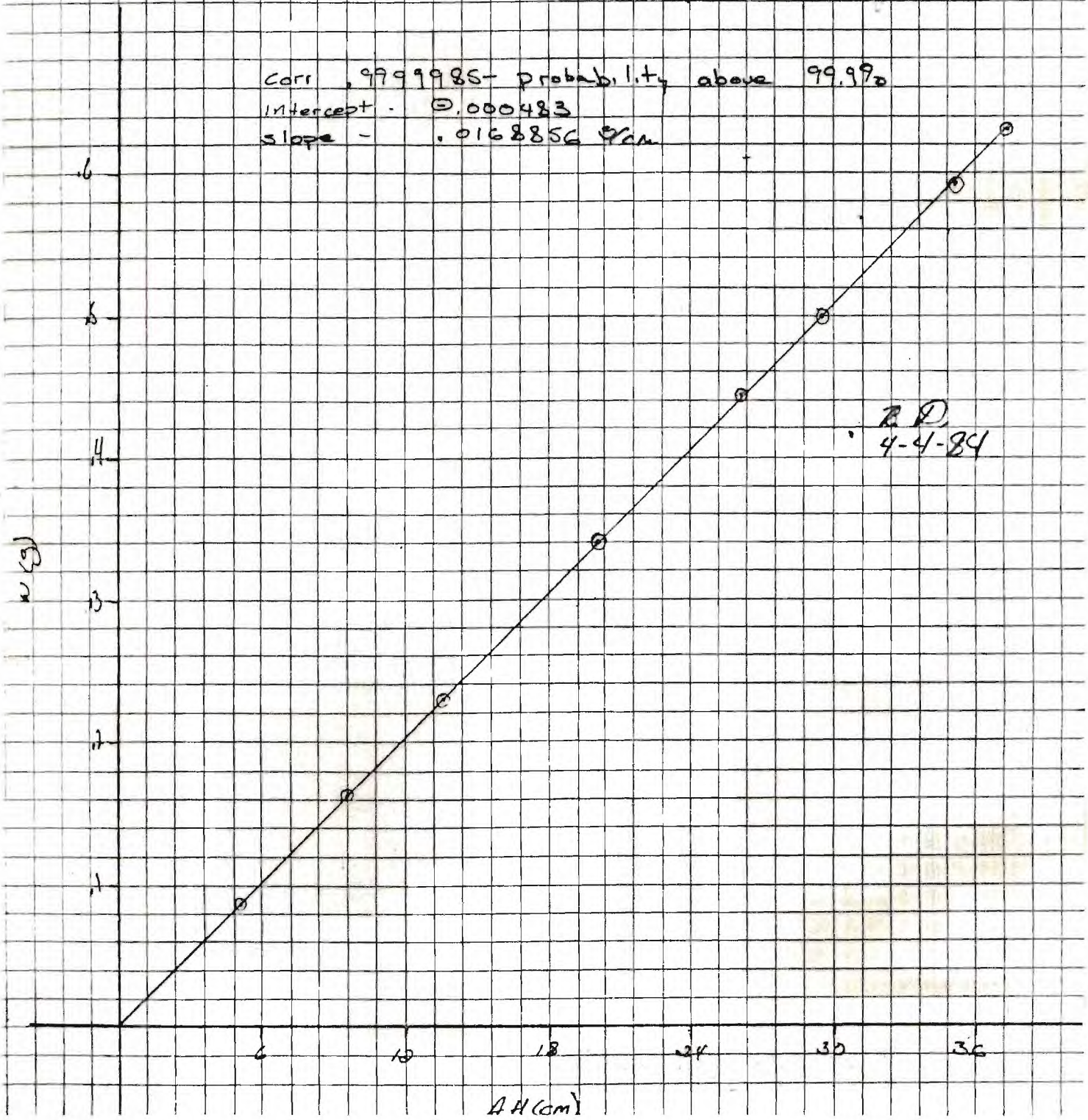


Fig. 8 Weight of Gas Adsorbed vs. Spring Displacement for tube 135

TABLE II. Zeolite Crushing Loads (kgf)

Hydrogen Type		AgZ Type	
5.36	8.86	5.81	1.57
3.03	12.36	1.60	0.70
7.02	2.62	0.92	0.75
5.95	4.47	5.34	0.69
3.05	3.22	0.51	5.72
4.08	6.50	2.00	6.43
5.08	2.78	4.83	6.50
1.98	2.30	0.59	1.85
10.28	9.93	0.18	1.14
5.63	2.89	2.91	8.25
5.83	17.04	0.20	9.57
4.75	13.50	2.18	3.90
6.00	3.63	0.94	0.99
3.49	5.60	2.99	4.71
7.14	5.61	0.80	6.86
2.84	2.74	5.60	0.50
2.70	11.84	0.62	0.20
6.81	5.21	2.21	1.58
9.39	5.60	0.19	1.71
2.48	7.36	3.52	1.00
1.86	8.79	2.18	2.36
7.44	9.41	17.14	1.37
5.64	6.80	0.59	0.96
8.59	2.04	10.44	2.14
4.18	5.97	0.43	1.58

$\bar{x}$	5.953	2.955
S	3.285	3.289
80% Spread	1.748 - 10.158	0.0 - 7.165
95% Reliability	5.042 - 6.864	2.043 - 3.867

Comparison of these crushing strengths using a two tailed t-test indicates that there is a highly significant difference in their crushing strengths. The samples tested were between 0.23 and 0.52 inches in length. There was not a significant correlation between length and crushing load. Tests were carried out according to ASTM D 4179. The applied rate of force was increased at 4.5 lbf/sec.

Designation: D 4179 – 82

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa. 19103
Reprinted from the Annual Book of ASTM Standards, Copyright ASTM
If not listed in the current combined index, will appear in the next edition.

Standard Test Method for SINGLE PELLET CRUSH STRENGTH OF FORMED CATALYST SHAPES[1]

This standard is issued under the fixed designation D 4179, the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers determining the resistance of formed catalysts to compressive force and is applicable to regular catalyst shapes such as tablets and spheres. Extrudates, granular materials, and other irregular shapes are specifically excluded.

1.2 This method determines the average crush strength in the range from 0 to 50 lbf (0 to 220 N).

1.3 The values stated in pound units are to be regarded as the standard.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable Document

2.1 *ASTM Standard*

E 177 Recommended Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material[2]

2.2 Other Document

ASTM STP 447 A, Manual on Test Sieving Methods[3]

3. Significance

3.1 This method is intended to provide information concerning the ability of a catalyst shape to retain physical integrity during use.

4. Description of Terms Specific to This Method

4.1 *tablets* tabletted cylindrical catalyst particles, either solid or hollow core, with lengths that do not vary from the mean by more than ±10%.

4.2 *pellets* any catalyst shape tablets, spheres, or other similar configuration, that are not otherwise excluded from the scope of this test.

5. Summary of Method

5.1 Individual pellets taken from a representative sample are placed between two flat surfaces, subjected to a compressive load, and the force required to crush the pellet is measured. The procedure is replicated and the average of all measurements taken is determined.

6. Apparatus

6.1 A suitable compression testing device is required, consisting of the following:

6.1.1 *Calibrated Gage*, marked for direct reading of the force in pounds (newtons). Additionally, a suitable system (mechanical, hydraulic, or pneumatic) must be provided so that the rate of force application is both uniform and controllable within specified limits.

---

[1] This method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D 32.02 on Physical Mechanical Properties.

Current edition approved Aug. 27, 1982. Published October 1982.

[2] *Annual Book of ASTM Standards*, Parts 13, 14, 15, 17, 25, and 41.

[3] Available from ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103.

6.1.2 *Tool Steel Anvils* between which the sample is to be crushed. The faces of the tool steel anvils shall be smooth and free from recesses or ridges that would interfere with uniform contact along the major axis of the pellet. When testing tablets or spheres, the anvils may be of any convenient size or shape as long as their length and width are greater than the corresponding dimensions of the tablet or pellet being tested (see Fig. 1).

7. Sampling

7.1 A test sample of 50 to 200 individual pieces shall be obtained from larger composites by riffling or splitting in accordance with STP 447A (Section 5.12) with the aim of obtaining a representative sample that represents shape and size distribution of the larger composite. The size of the sample shall depend on the precision required and the homogeneity of the material being tested.

7.2 Heat the test sample(s) at 400 ± 15°C for not less than 3 h. Normally, this treatment can take place in air, however, in the case of materials that might react with air at elevated temperatures (such as prereduced catalysts) the heat treatment should take place in an inert atmosphere.

7.3 After heating, cool the test sample(s) in a desiccator or other suitable container to eliminate the possibility of moisture adsorption prior to testing.

NOTE 1—Since many catalyst formulations are strong adsorbents, the use of 4A (cobalt-treated) molecular sieves as a desiccating medium is suggested. Regenerate the desiccant at 220 to 260°C, as required.

8. Calibration and Standardization

8.1 Prior to use, set the test apparatus to zero and calibrate with any commercially available force gage with marked graduations of no more than ½ lbf (2 N) and having accuracy traceable to the National Bureau of Standards, or other similar authority.

9. Procedure

9.1 Remove from the desiccator only that number of pellets that can be tested within a 10-min period.

NOTE 2—Precaution must be taken to assure that moisture pick-up in the 10-min period will not significantly affect the pellet crush strength.

9.2 Place a single-catalyst pellet between the anvils of the compression testing device. Orient each pellet in the same direction before crushing. For those pellets capable of being tested in different orientations, report the one used. Figure 1 shows pellets in radial and axial orientations. Use tweezers, forceps, or other suitable device or procedure to prevent the transfer of moisture from the operator's hands to the piece being tested.

9.3 Apply increasing force at a uniform rate in the range of 1 to 10 lbf/s. (4.4 to 44 N/s.) until the pellet crushes or collapses. Compression of surface irregularities or limited fracturing of a pellet followed by continued resistance to increasing load are not to be used as criteria for determining the end-point of this test.

9.4 Read and record, to the nearest one-half graduation, the force indicated on the calibrated dial of the apparatus at the instant of collapse.

9.5 Separate the anvils and remove all residue with a soft cloth or brush. Ensure that the faces of the anvils are free from adhering particles.

9.6 Repeat Steps 9.2 through 9.6 until all pellets in the sample have been crushed. Record the crush strength for each pellet tested.

10. Calculations

10.1 Calculate the average crush strength ($\bar{X}$) retaining one more decimal place than the recording values as follows:

$$\bar{X}\ \text{lbf (N)} = (\Sigma X)/(n)$$

where:

ΣX = the sum of all observed crush strengths and

n = the number of pellets crushed.

10.2 Calculate the standard deviation of the n readings to 3 significant digits as follows:

$$S = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n - 1}}\ \text{lbf (N)}$$

where:

S = standard deviation of the individual strength values and

$\Sigma(X - \bar{X})^2$ = sum of the squares of the deviations of each recorded reading from the average strength.

NOTE 3—Many calculators are programmed to perform these operations and to report average and standard deviation directly. It is important to verify that the program chosen uses the $n - 1$ denominator rather than n in calculating standard deviation.

11. Report

11.1 Report the average crush strength to one more decimal place than the recorded data on the individual strengths. For pellets capable of being tested in different orientations, the one used should be reported.

11.2 Report the "80 % spread" (that is, the range within which 80 % of the individual pellet strengths are expected to fall, assuming that individual pellets form a "normal" distribution). Calculate as follows:

$$80\ \%\ \text{spread} = \bar{X} \pm 1.28S$$

11.3 Report the "95 % Reliability" of the average reported in 11.1. This is the uncertainty inherent in the reported average, expressed as the range within which 95 % of the averages of an infinite number of test samples of n pellets would be expected to fall were they to be similarly drawn from the same lot and tested. Calculate as follows:

$$95\ \%\ \text{reliability} = \bar{X} \pm 1.96\ S/\sqrt{n}$$

11.4 Report the applied rate of force increase, if available.

12. Precision and Accuracy[1]

12.1 The precision is based on the results of a multilaboratory multisample study, and the procedures and definitions of Recommended Practice E 177 were used.

12.1.1 *Repeatability*—The single-laboratory, multiday repeatability is ±7 % (2*S* %) of the mean measured value for spheres and ±8 % (2*S* %) of the mean measured value for tablets.

12.1.2 *Reproducibility*—The multilaboratory, multiday reproducibility is ±9 % (2*S* %) of the mean measured value for spheres and ±19 % (2*S* %) of the mean measured value for tablets.

12.2 All of these statements apply to "test results", each of which is the mean of 100 crush strength measurements, and are based on levels of 20 lbf for ⅝ in.-spheres and 16 lbf for ½ in. tablets.

12.3 An estimate of the accuracy of the method is not possible.

[1] Supporting data are available from ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103 and may be obtained by requesting RR: D32-1004.

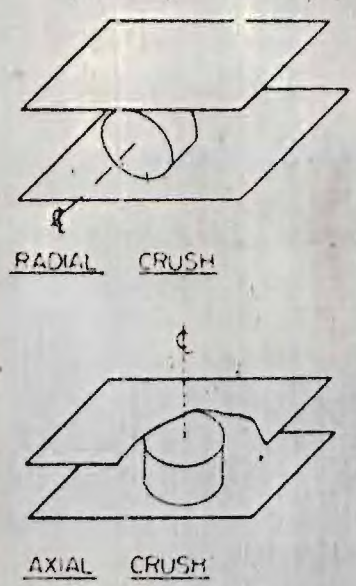


FIG. 1 Radial and Axial Crush

AG0-602

Georgia Institute of Technology
SCHOOL OF NUCLEAR ENGINEERING AND HEALTH PHYSICS
ATLANTA, GEORGIA 30332

NEELY NUCLEAR RESEARCH CENTER

(404) 894-3600

August 16, 1984

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This letter is a progress report for the month of July, 1984 on the project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses".

1. Zeolite Crushing and Hardness Tests

As reported last month, the ASTM Standard D-4179-82 for crushing test requires that the sample be heated to 400°C for not less than 3 hours. We did not know, if in the process of heating the sample, the resistance to crushness changes. Consequently, another sample, without being heated was measured. The results are given in Table I. It is seen that for the silver zeolite sample, heating affects the sample. Consequently, unless told otherwise by SRL, we will not heat samples prior to crushness tests.

Procedures for the hardness test have now been devised. Details and results will be reported next month. The problem with the hardness test was that the zeolite pellets are weak and ASTM Standard tests could not be applied.

2. Zeolite Density Determination

Pellets will be broken into lengths no longer than 1/4 inch. Approximately two grams of each material will be dried at 400°F for four hours and then cooled in a desicator. After cooling, the sample will be weighed to the nearest 0.0001 grams. It will then be soaked in kerosene for 24 hours to saturate it. The weight of the sample suspended in kerosene will then be determined to the nearest 0.0001 grams.

$$\text{Volume} = \frac{\text{Weight in air} - \text{Weight suspended in kerosene}}{\text{Kerosene density}}$$

$$\text{Sample density} = \frac{\text{Weight in air}}{\text{Volume}}$$

Triplicate determinations will be made on each sample and both the individual and average values reported.

3. Sample Holders Leak Tests

Helium leak tests of all sample holders revealed that small leaks occur in all the SS-4H valves purchased from the Georgia Valve and Fitting Company. The Company was contacted; the problem was traced to the degreasing step in which the sample holders were degreased by trichlorethylene vapor. This step apparently dissolved the grease between the valve handle and the valve body. This grease is only needed for the proper closing of the valve. The seal in the valve is metal to metal contact; no grease is needed. The problem of valving has now been solved.

4. Gas Analysis

Detailed procedures for the gas analysis are being developed using mass spectrometry. These procedures will be submitted to SRL for approval.

This is basically where we are on this project. If you have any questions please let me know.

With best wishes.

Sincerely,

R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Table I

Zeolite Crushing Loads (kgf)
(Undried and Unheated)

Hydrogen Type		AgZ Type	
4.47	8.82	4.90	2.25
5.26	2.17	6.06	17.62
2.83	2.78	5.30	10.76
5.20	4.24	5.13	1.65
7.17	2.59	8.00	2.30
2.33	4.15	5.97	5.89
4.62	2.60	14.89	9.12
9.72	4.79	4.81	1.66
5.68	5.41	4.02	4.32
8.60	1.97	0.52	4.50
3.36	3.55	3.53	2.41
8.01	2.44	1.20	0.62
4.95	5.51	1.71	0.90
4.63	5.04	6.41	2.84
7.96	1.49	2.20	0.83
6.26	2.27	1.94	12.36
8.84	1.75	2.84	11.64
5.20	5.52	3.53	10.04
8.46	2.52	4.63	4.87
6.08	3.72	6.94	0.71
2.25	9.77	2.51	1.72
6.87	8.93	2.63	1.60
8.84	4.88	12.52	1.01
3.90	2.57	2.11	1.31
3.22	2.94	8.52	0.53

$\bar{x}$	4.943	4.726
s	2.384	4.040
80% Spread	1.891 - 7.994	0.0 - 9.897
95% Reliability	4.282 - 5.604	3.606 - 5.846

Comparison of these crushing strengths and those of the dried samples using a two tailed t-test indicates that the only significant difference was between the dried and undried AgZ samples. The samples tested were between approximately 0.25 and 0.50 inches in length. These tests were carried out according to ASTM D 4179 except the samples were not dried. The applied rate of force was increased at 4.5 lbf/sec.

A-60-602

Georgia Institute of Technology
SCHOOL OF NUCLEAR ENGINEERING AND HEALTH PHYSICS
ATLANTA, GEORGIA 30332

NEELY NUCLEAR RESEARCH CENTER

(404) 894 3600

October 2, 1984

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This letter is a progress report for the months of August and September, 1984 on the project entitled "Characterization of Changes in Adsorbent Properties Under High Radiation Doses." Last week I attempted to reach you by telephone on numerous occasions without success. I wanted to give a verbal report.

I. Procedures for Zeolite Crushness, Hardness, and Density Measurements

The procedures for crushness and density measurements have been reported in previous monthly reports. The hardness procedures and results on hydrogen and silver zeolite samples are given in Table I. Also included in Table I are the values for the density of the two zeolites

II. Sample Holder Leak Tests

All sample holders have been tested for leaks. Forty four of the 48 sample holders were tested by helium leak detector. Four holders equipped with pressure gauges could not be evaluated by helium leak detection because of possible damage to the pressure gauges. These holders were filled with air to a pressure of 30 psi and the pressure was noted daily for the past 30 days. The results show that all 48 sample holders are now leak proof.

III. X-Ray Diffraction and Oxygen Adsorption Measurements

The procedures for these measurements were devised sometime ago and are ready to use, pending resolution of the gas analysis problem.

Air + 1000 ppm NO_2

Air + 1000 ppm NO_2 + 1000 ppm O_3

This analysis is scheduled for the first week of October. I will keep you informed.

Best wishes.

Sincerely,

Ratib A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Enclosures

P.S. The gentleman I talked with at North Carolina is Dr. Altschuller (919) 541-2191).

IV. Gas Analysis

For the last two months we have spent about two-man months attempting to quantitatively analyze premixed gasses by mass spectrometry. Due to the fact that there are no standards of gas mixture containing NO_2, O_3, moisture, and air, even at the National Bureau of Standards, the mass spectrometry method is proving to be a good detection method, i.e., we are able to identify the components in the mixture but not quantitatively. The problem is in part due to the gas mixture itself. This mixture seems to be very active and is affected by light, temperature, and surface conditions. According to Dr. D.D. Davis (renowned expert in this area) the following reactions take place:

$$NO_2 + h\nu \longrightarrow NO + O$$

$$O + O_2 \longrightarrow O_3$$

$$3NO_2 + O_3 \longrightarrow 3NO_3$$

$$NO_3 + NO_2 \longrightarrow N_2O_5$$

$$NO_3 \longrightarrow NO + O_2$$

$$NO + O_3 \longrightarrow NO_2 + O_2$$

$$H_2O + NO_2 \longrightarrow HNO_3$$

Typical mass spectrometer data are appended. We have used low resolution and high resolution mass spectrometers. The conclusions from both measurements are that we can detect components of the air mixture but cannot quantify the amount without calibration of the spectrometer, which is invariably done with a standard gas (NBS) which we are not able to get.

In order to solve this problem we enlisted the help of Dr. D.D. Davis. Dr. Davis is able to quantify the NO_x with extreme precision (in parts per trillion). He accepted my request for analyzing initially two samples of the gas mixture as follows:

TABLE I. DENSITY AND HARDNESS MEASUREMENTS
(Untreated Zeolite Samples)

Density (gms/cm^3)

Hydrogen	Silver
1.8548	2.3255
1.8601	2.3259
1.8497	2.3243
$\bar{x}$ = 1.8549	$\bar{x}$ = 2.3252

Microhardness (Knoop Hardness Number)*

Hydrogen		Silver	
92.0	89.8	74.1	22.7
53.5	143.8	52.1	21.9
95.3	91.0	58.8	19.5
106.8	63.3	89.0	28.1
57.2	86.0	12.4	20.6
73.8	78.1	17.3	14.4
135.0	80.2	61.8	31.3
194.1	127.0	42.5	24.1
102.9	110.8	32.7	65.2
235.1	93.6	42.5	29.2
$\bar{x}$ = 105.5		$\bar{x}$ = 38.0	
S = 44.7		S = 21.9	

*These tests were done on a Tukon Tester manufactured by Wilson Mechanical Instrument Division of American Chain and Cable Company using a 175 gram load. The bottom of the samples were flattened on 180 grit SiC paper so they were stable on the testing stage. The tops of the samples were very lightly ground on 600 grit SiC paper before the hardness measurements were made.

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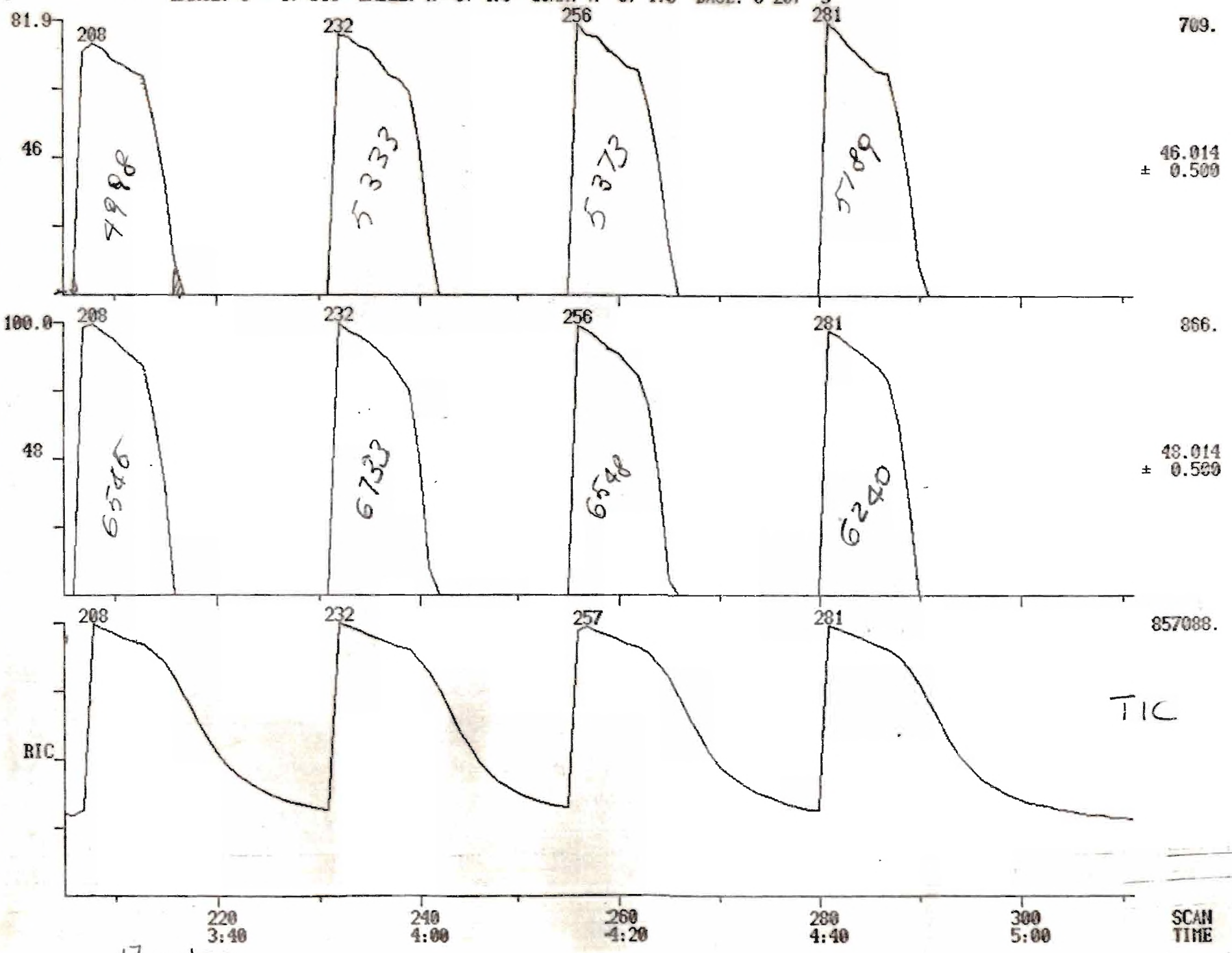

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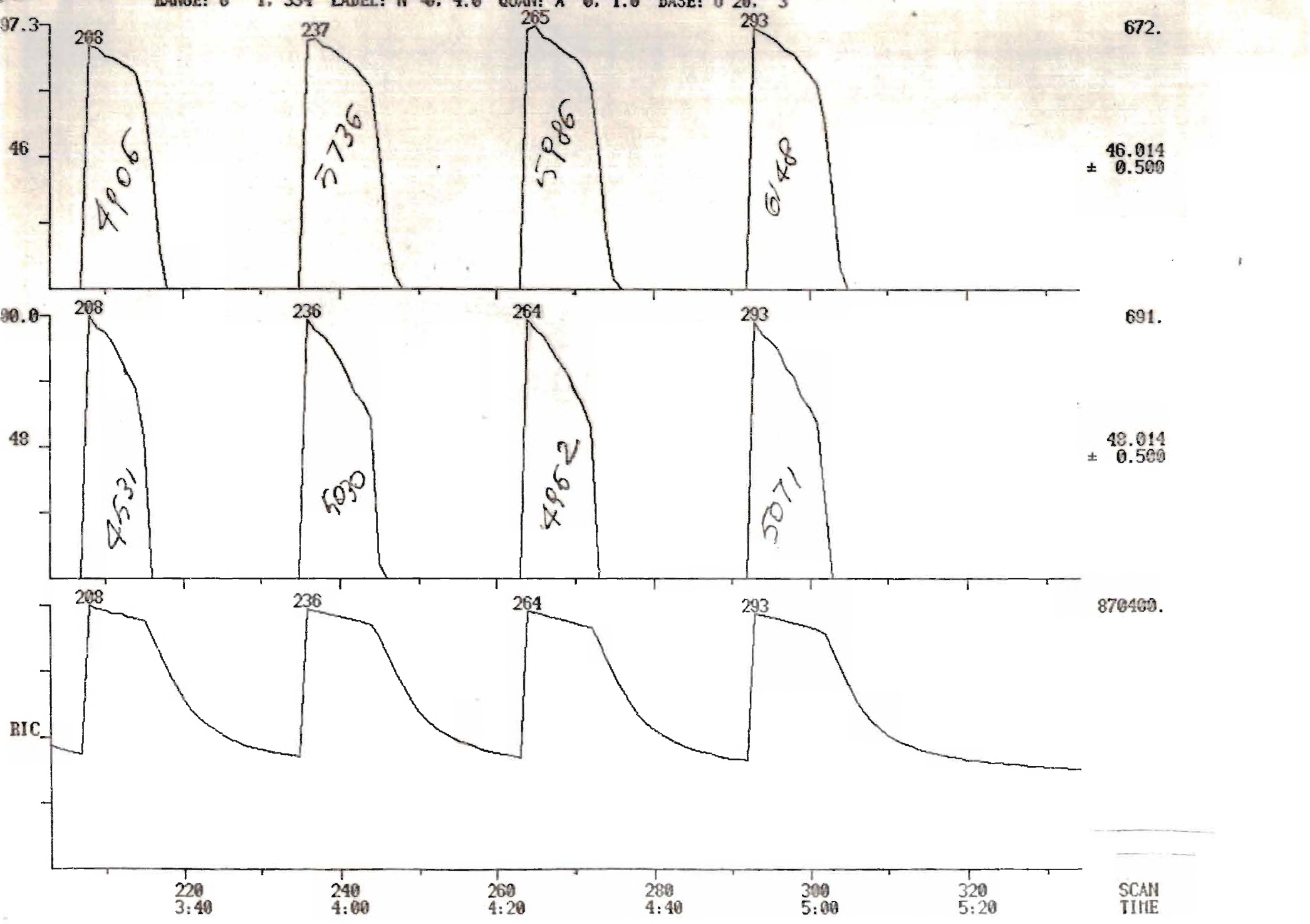
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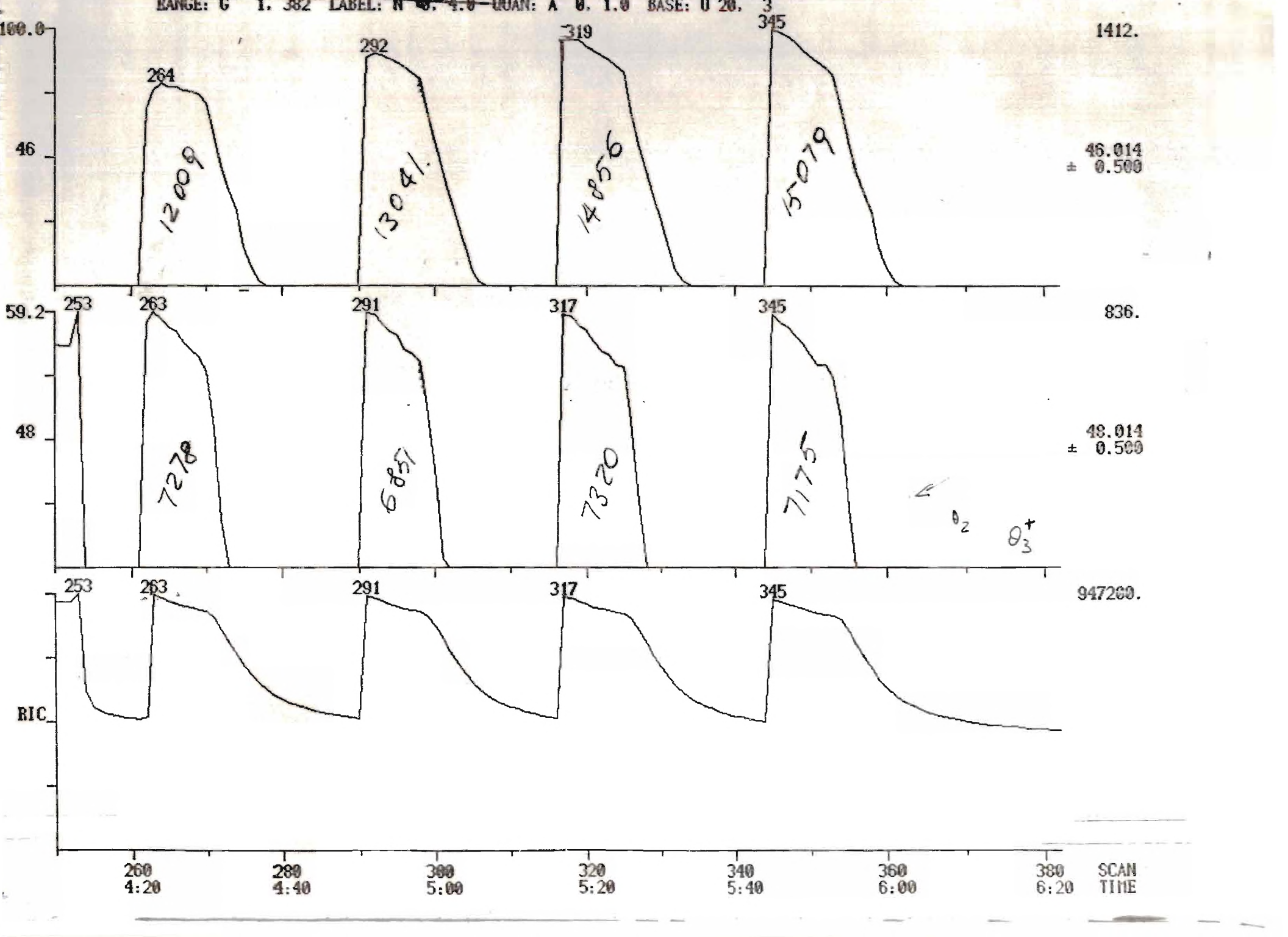
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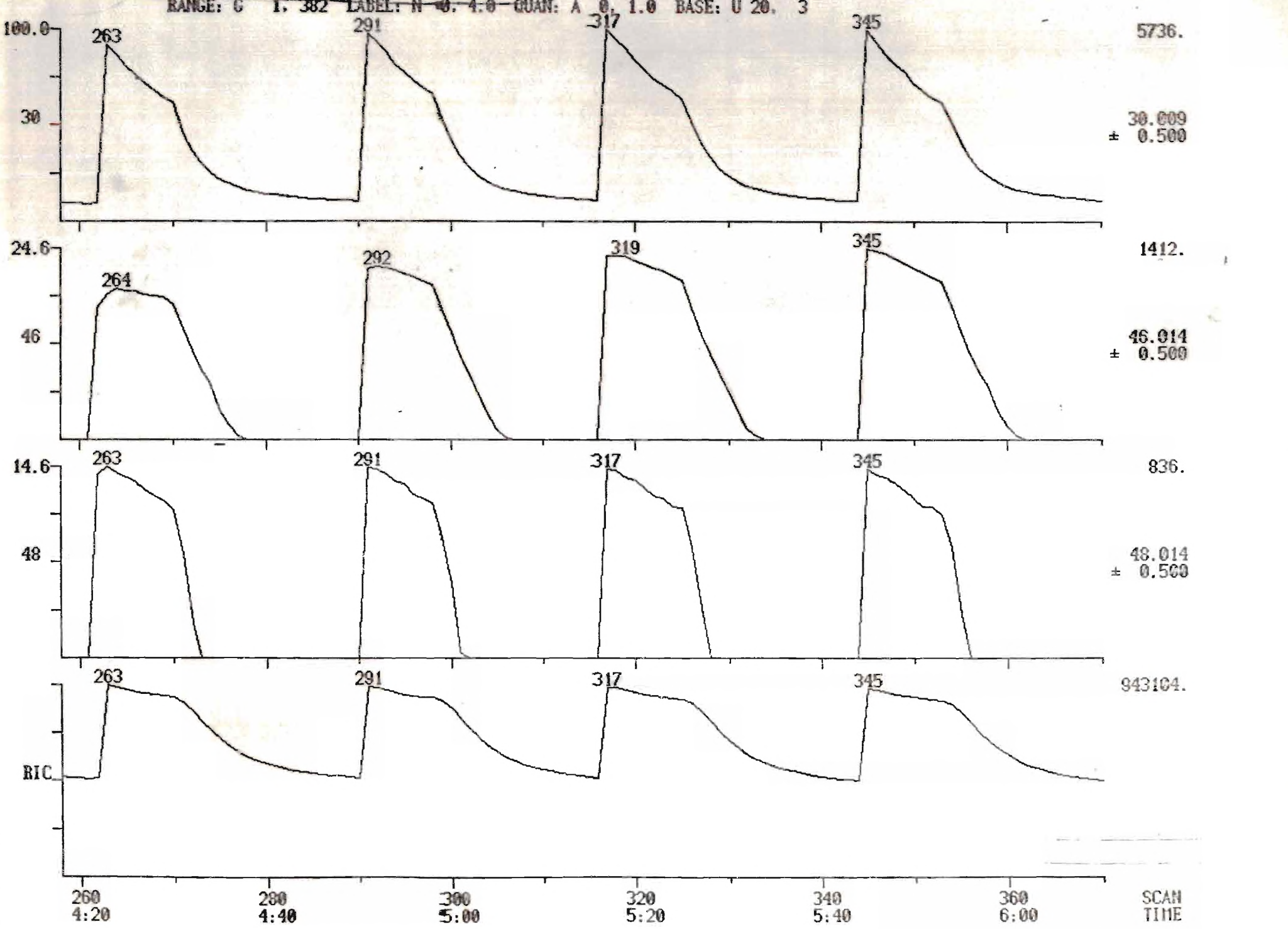
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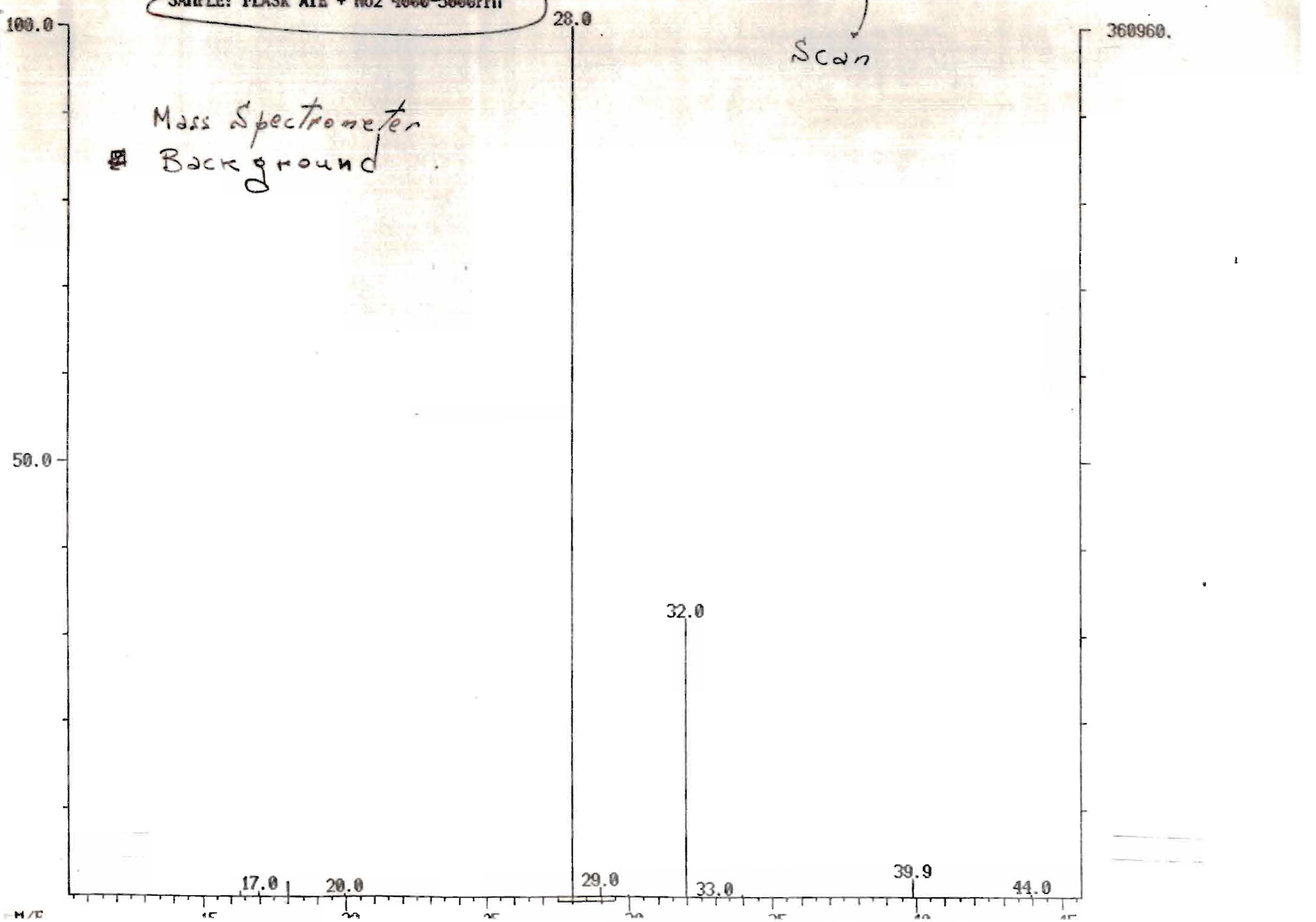
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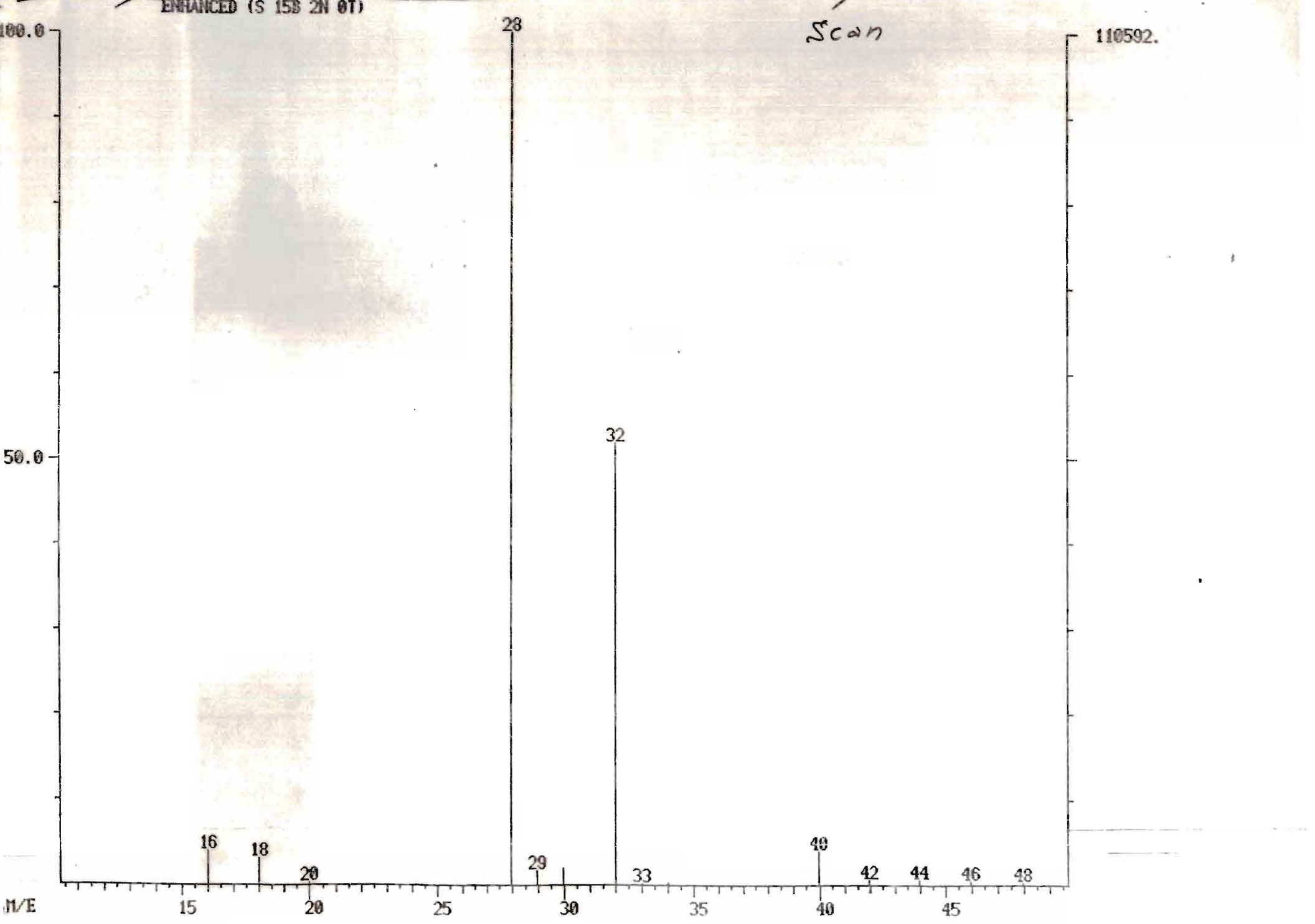

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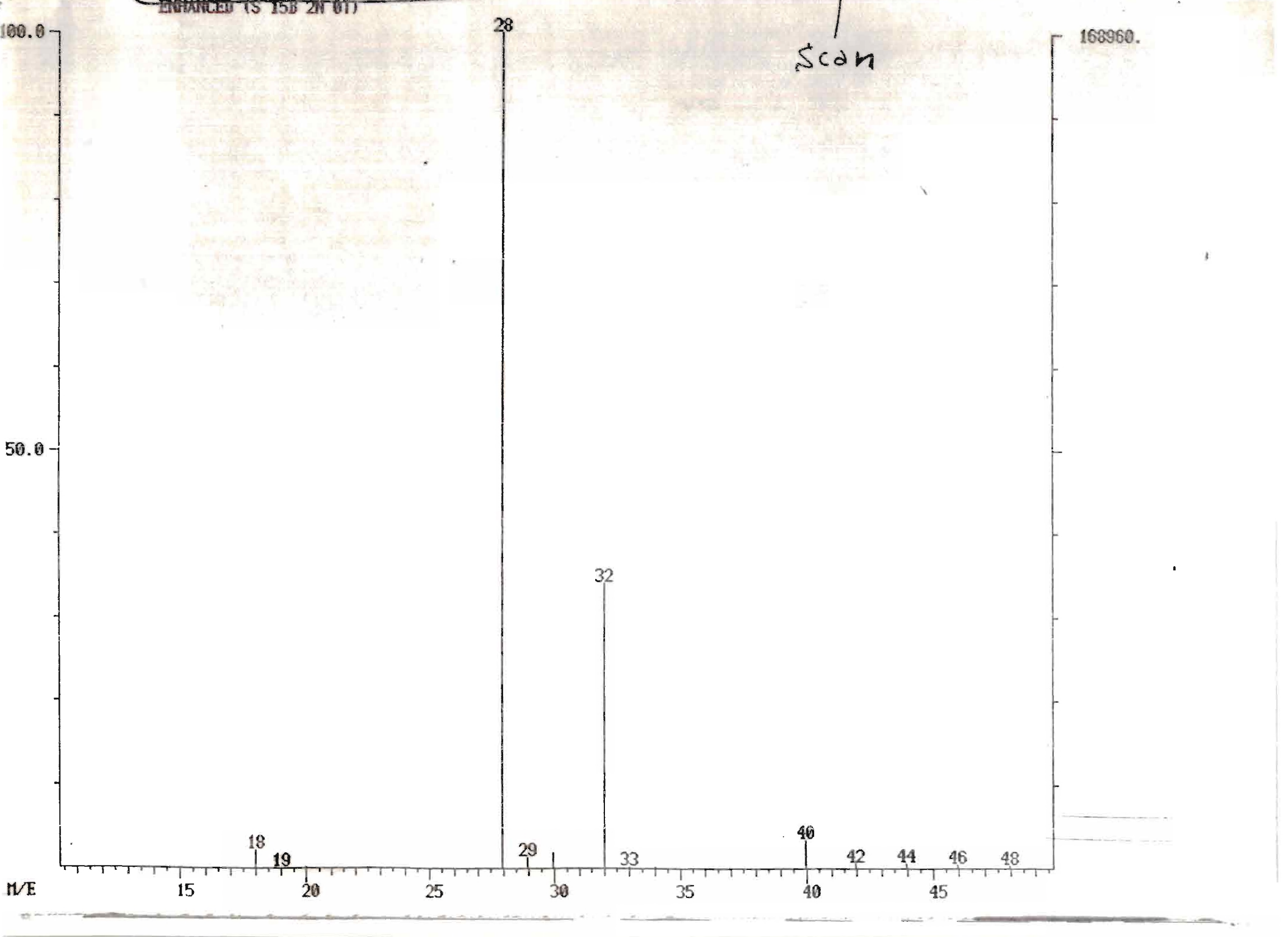

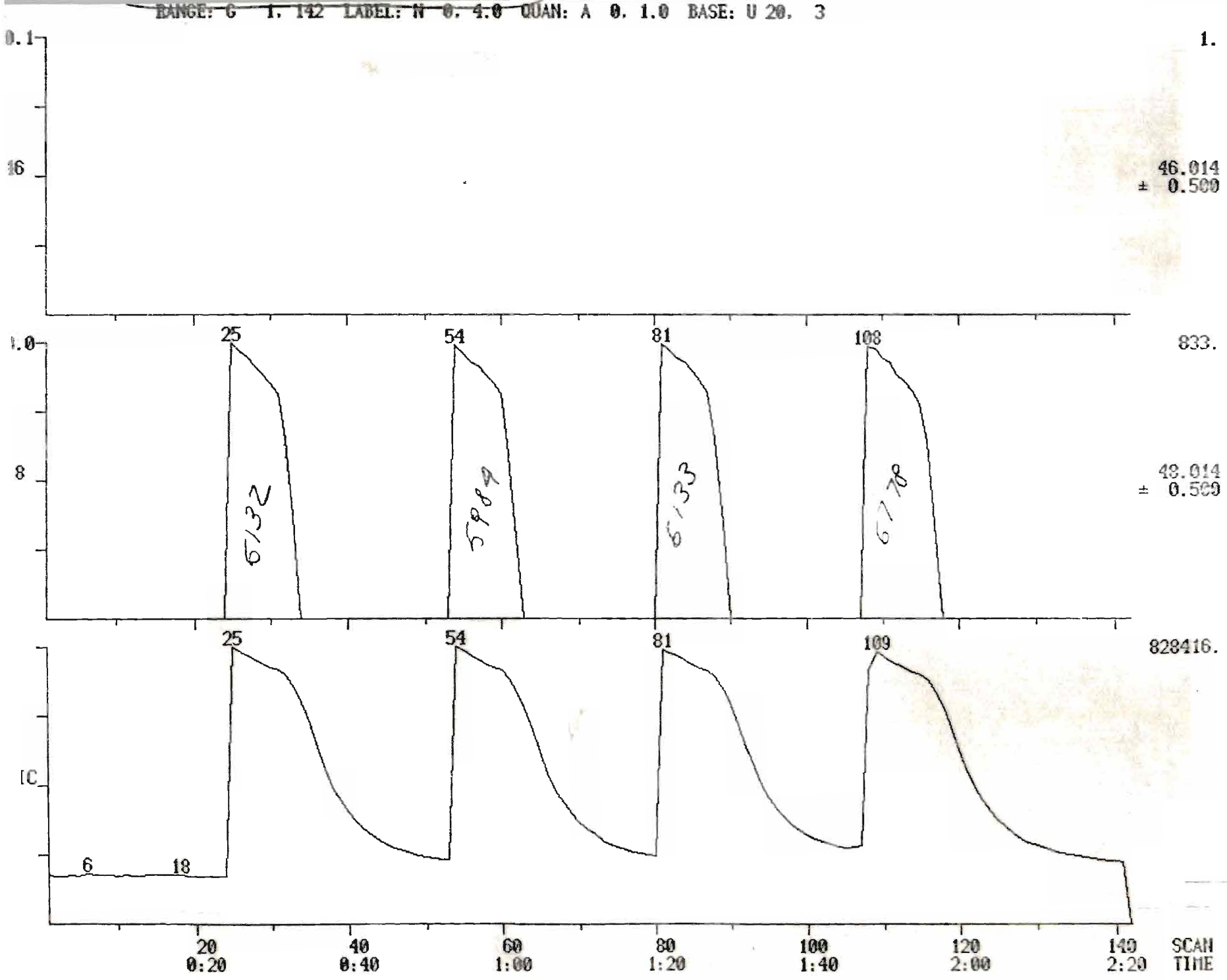
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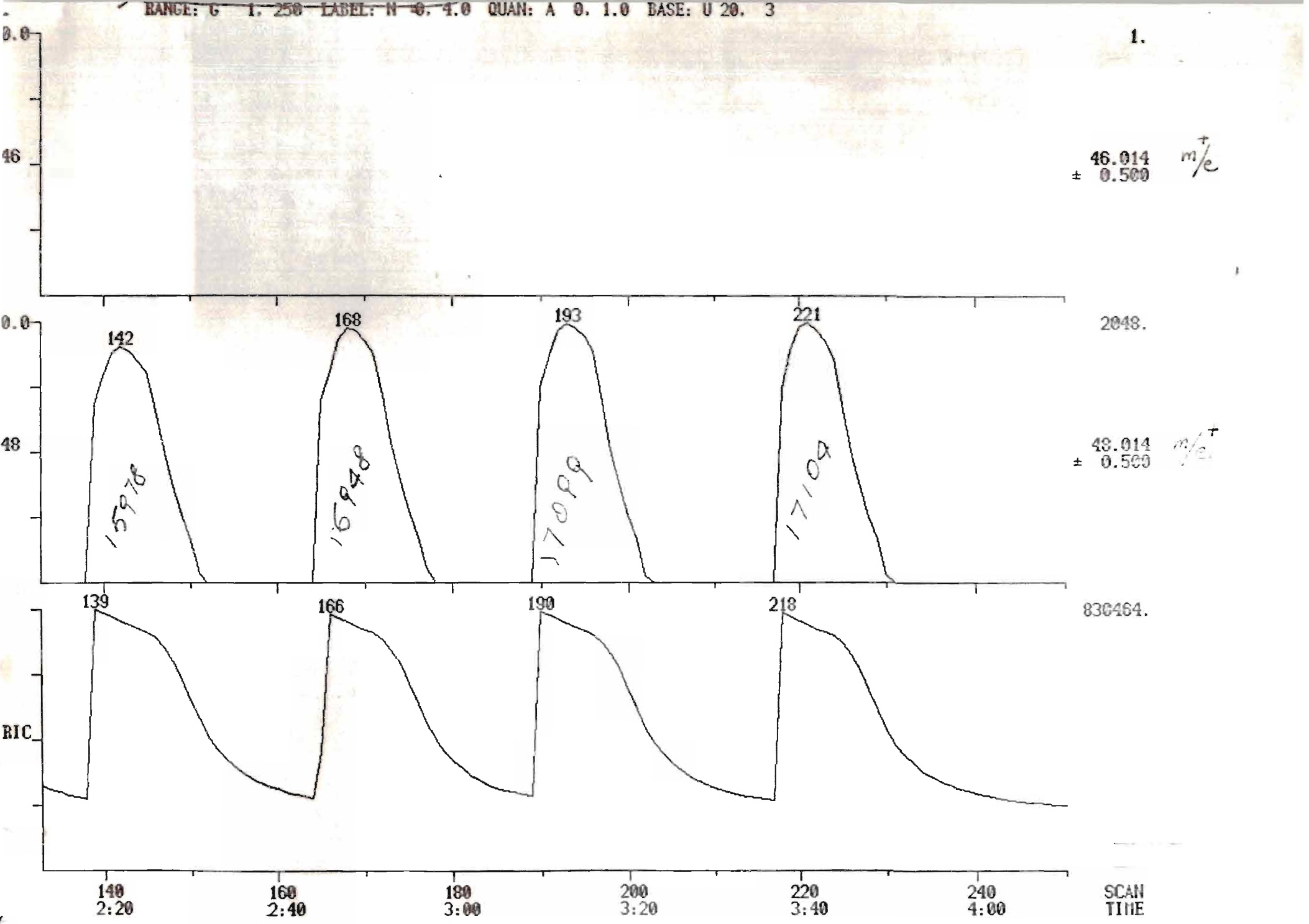

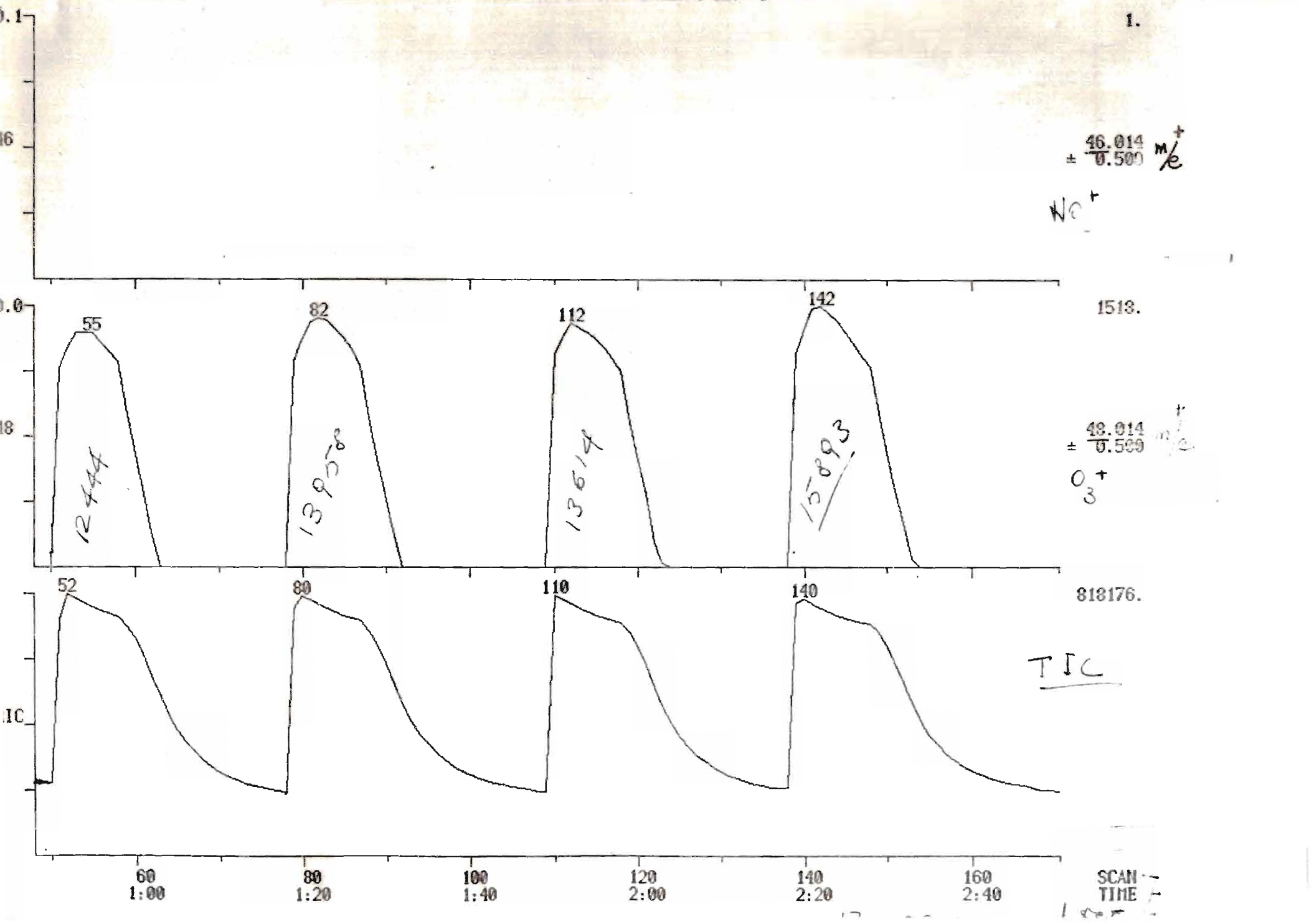
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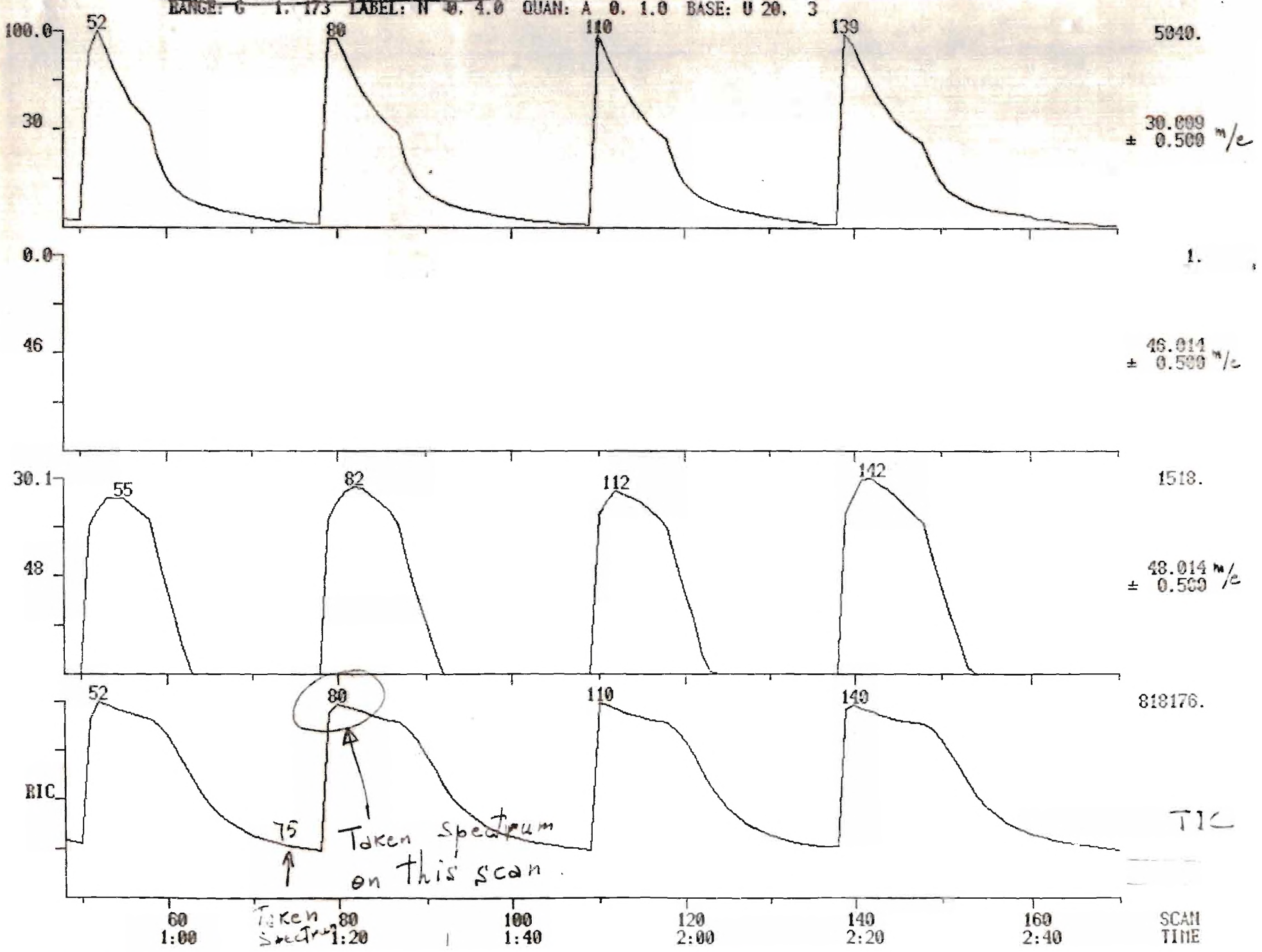
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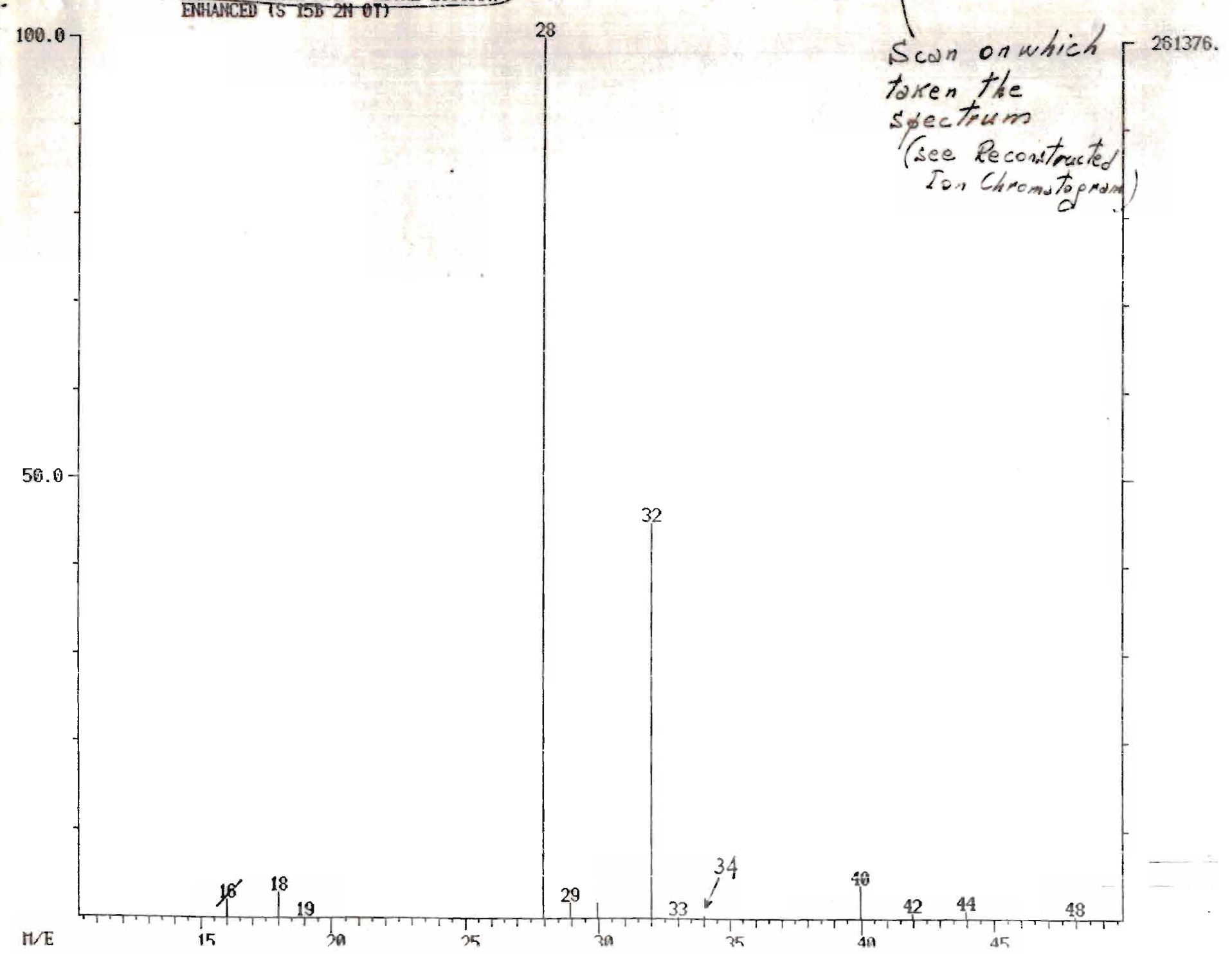

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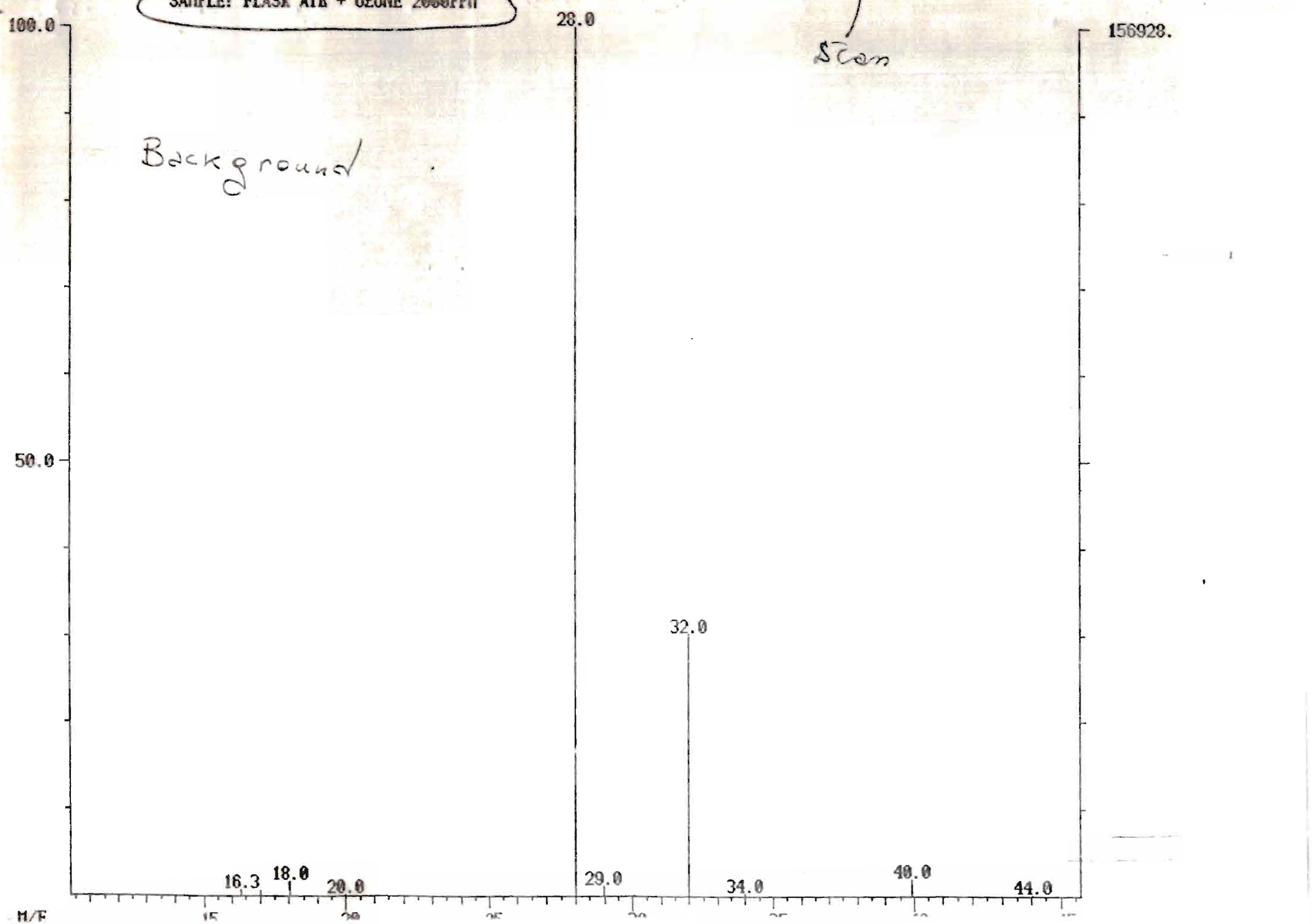

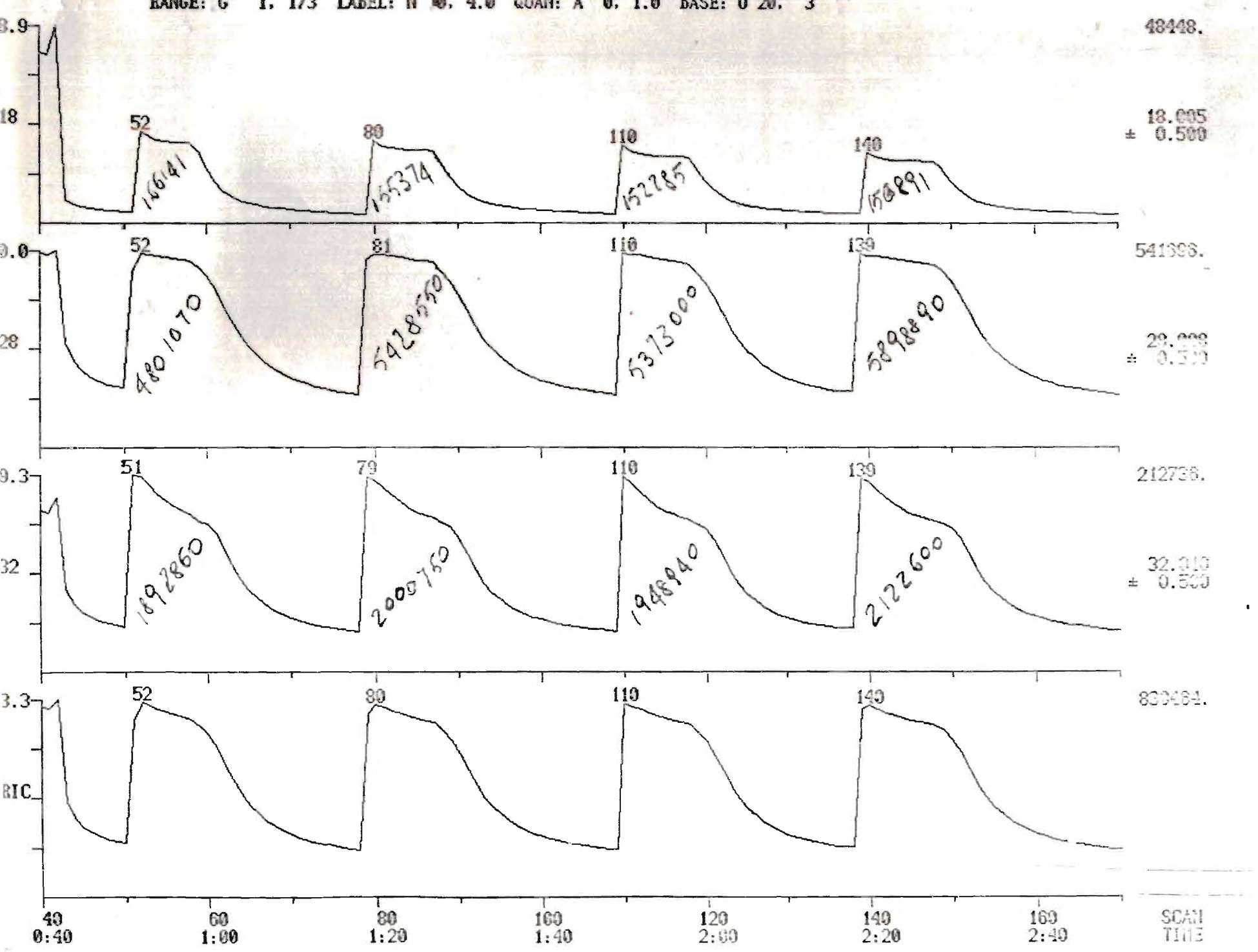
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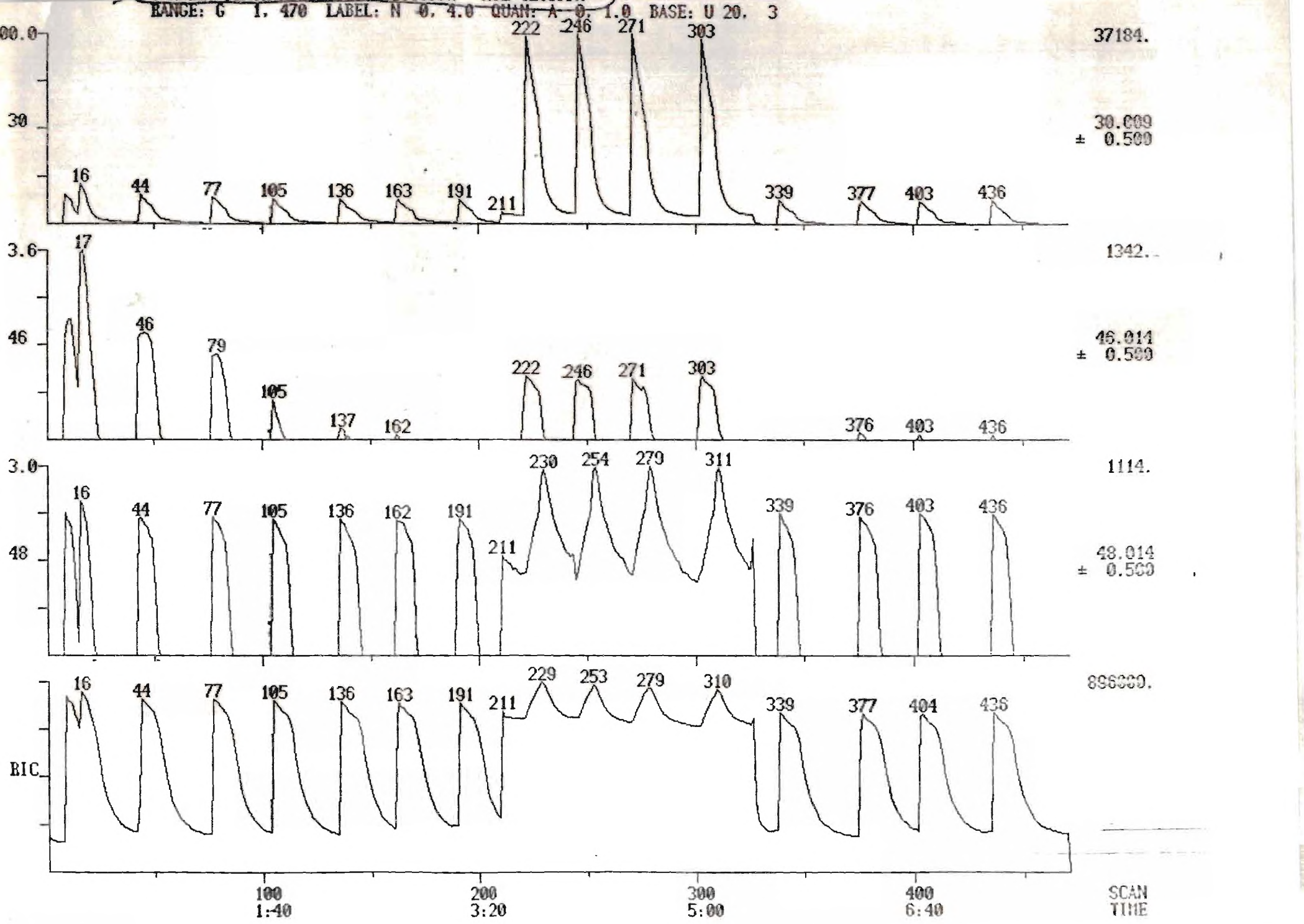
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16 44 77 105 136 162 191 211 230 254 279 311 339 376 403 436
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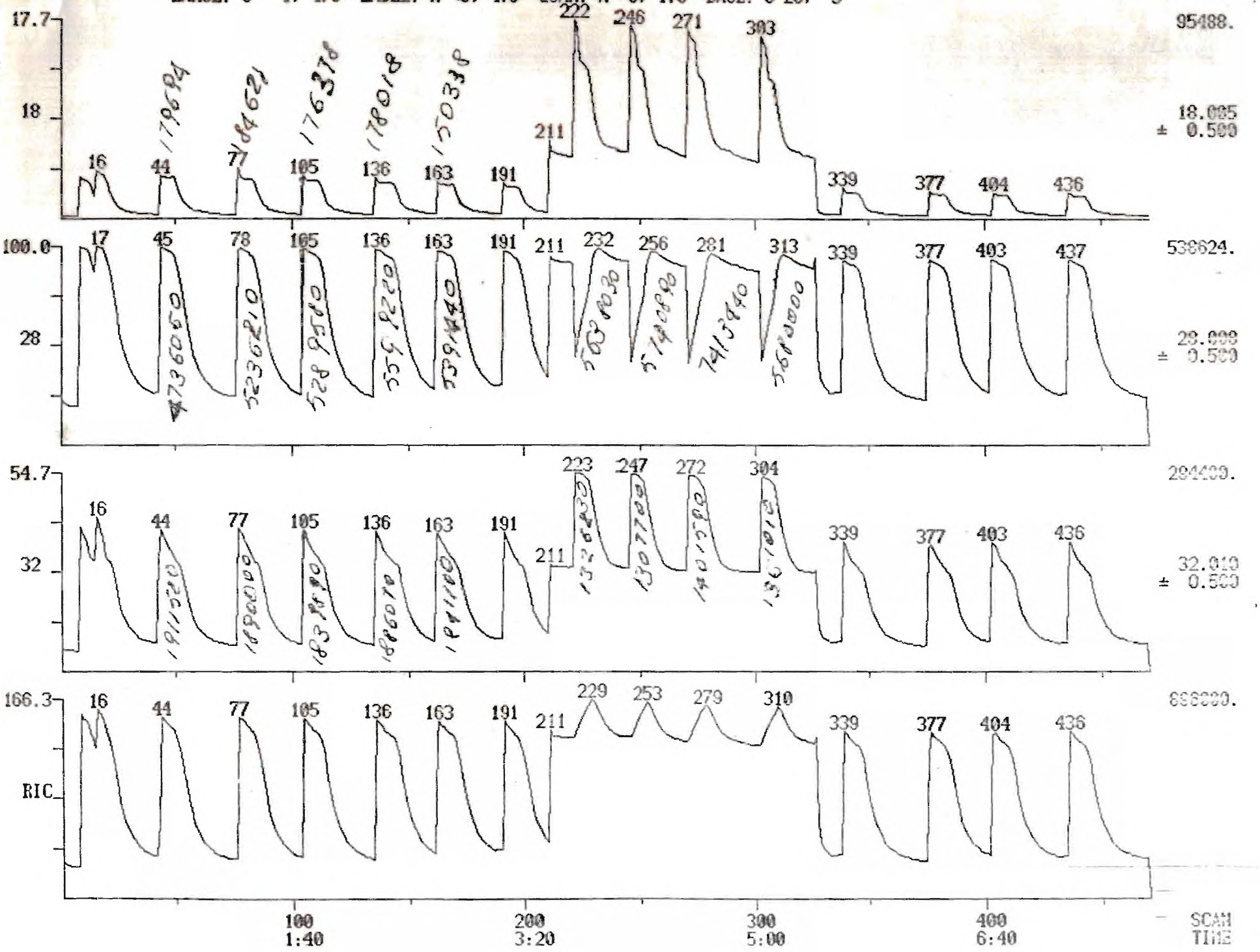
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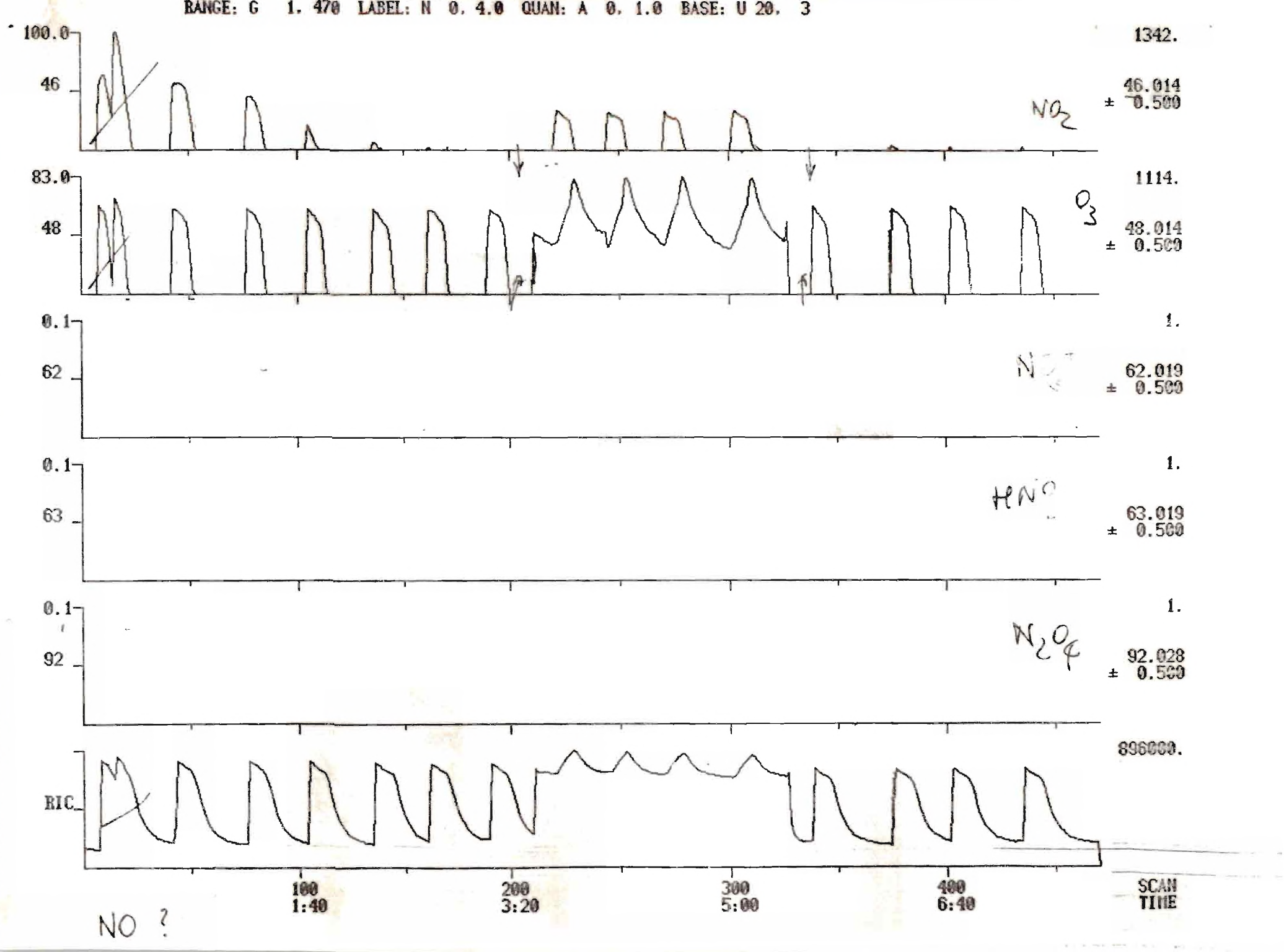
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1114.
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± 0.500
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63
1.
63.019
± 0.500
HNO
0.1
92
1.
92.028
± 0.500
N2O4
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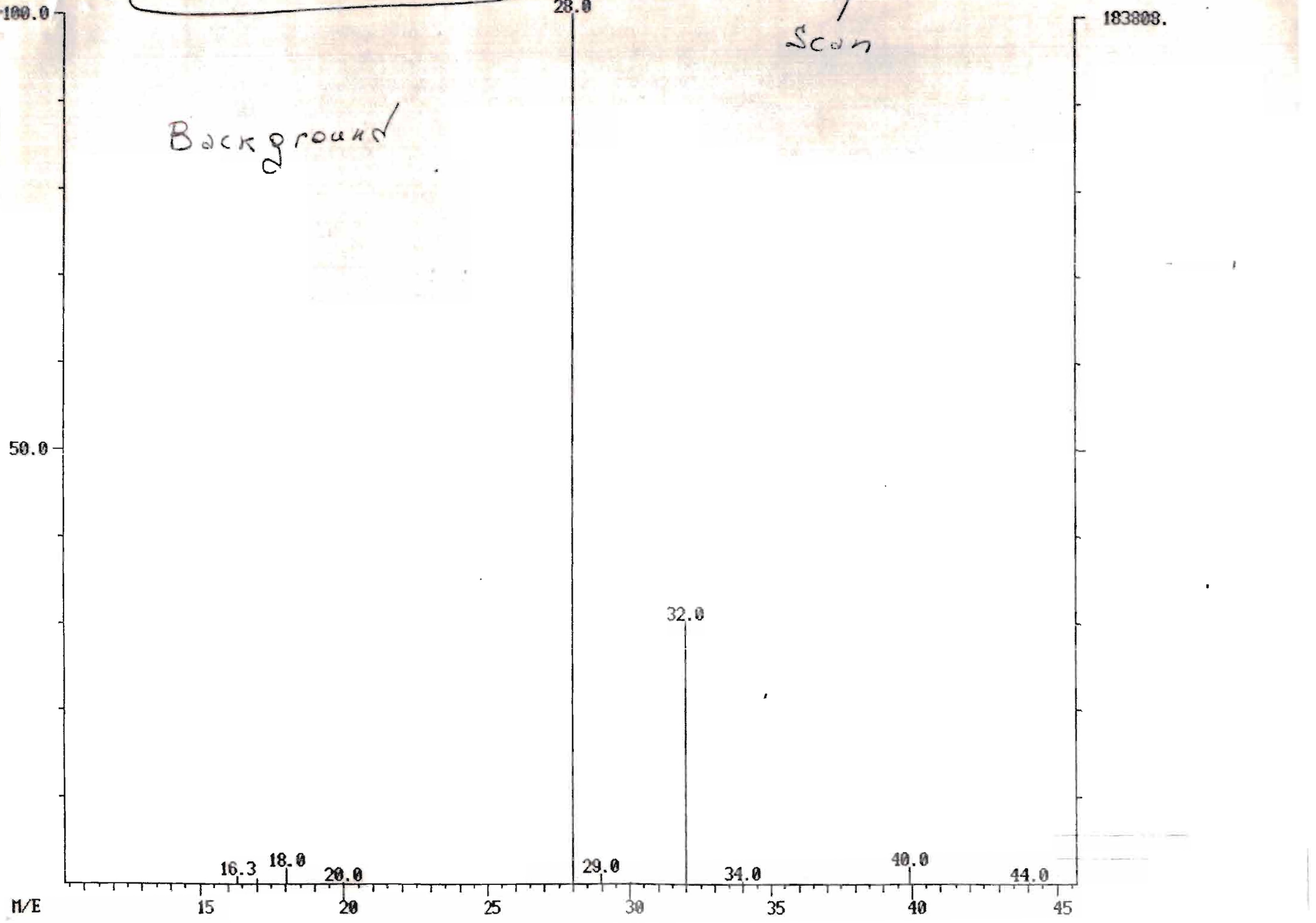
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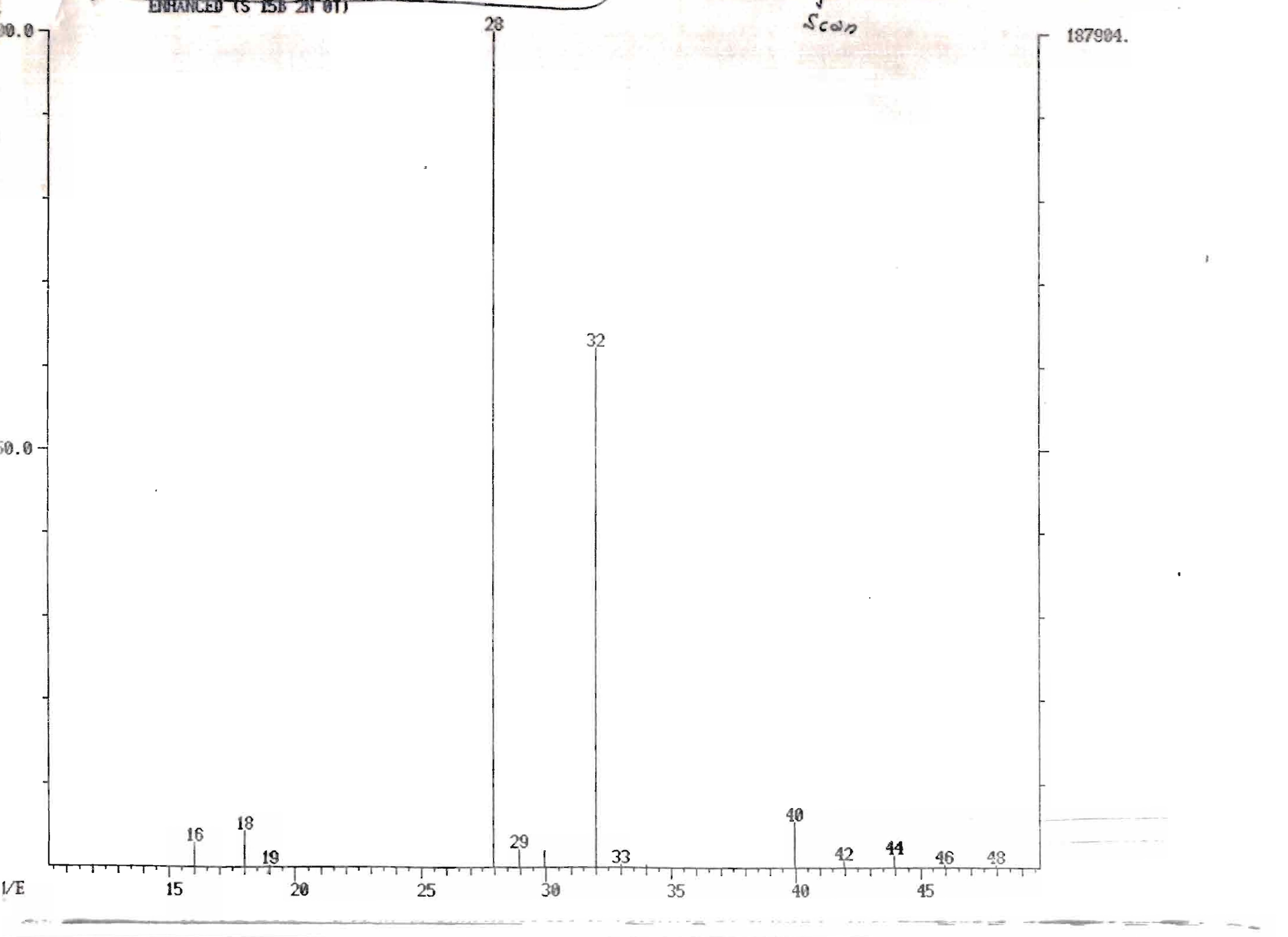

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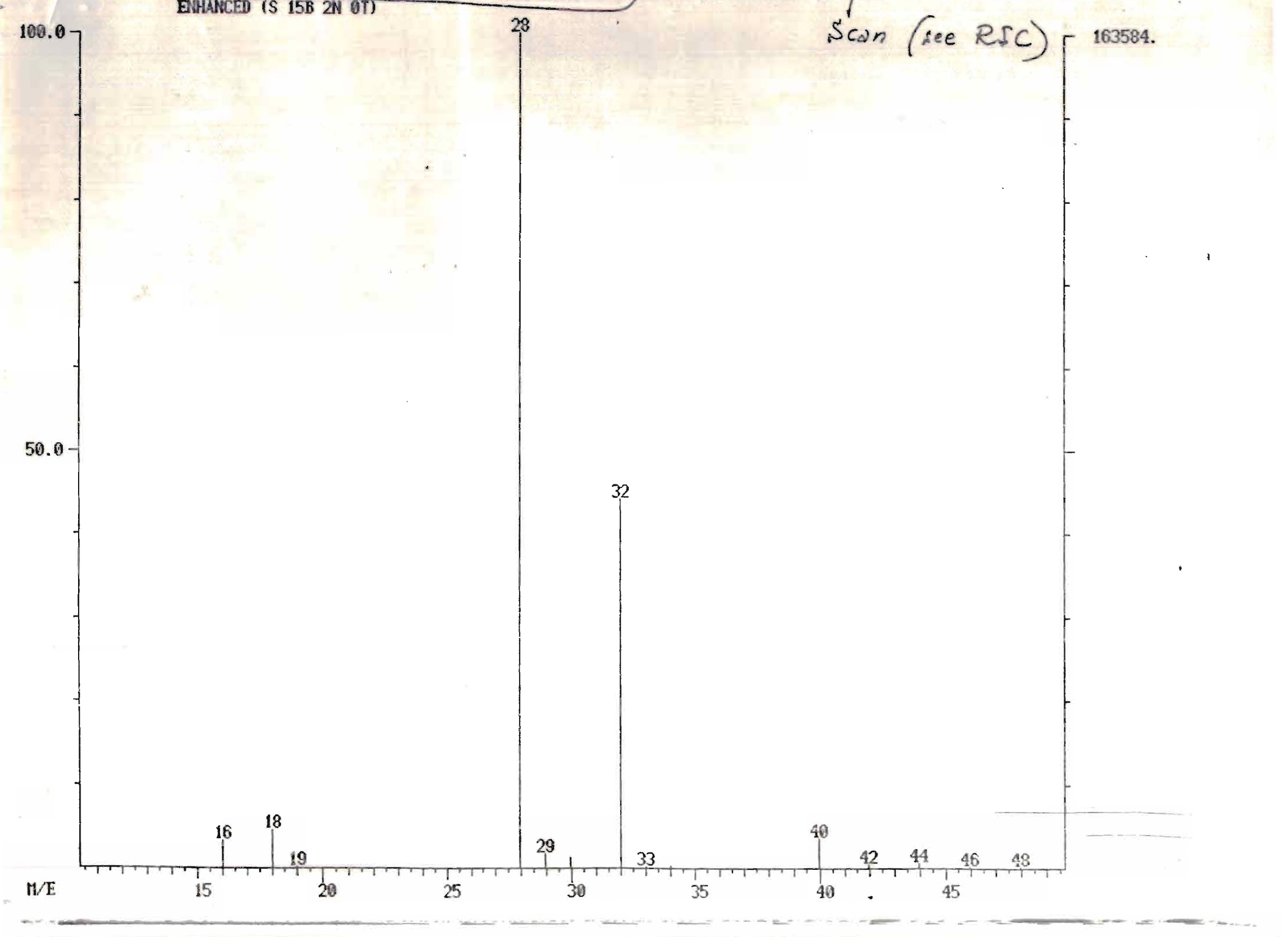

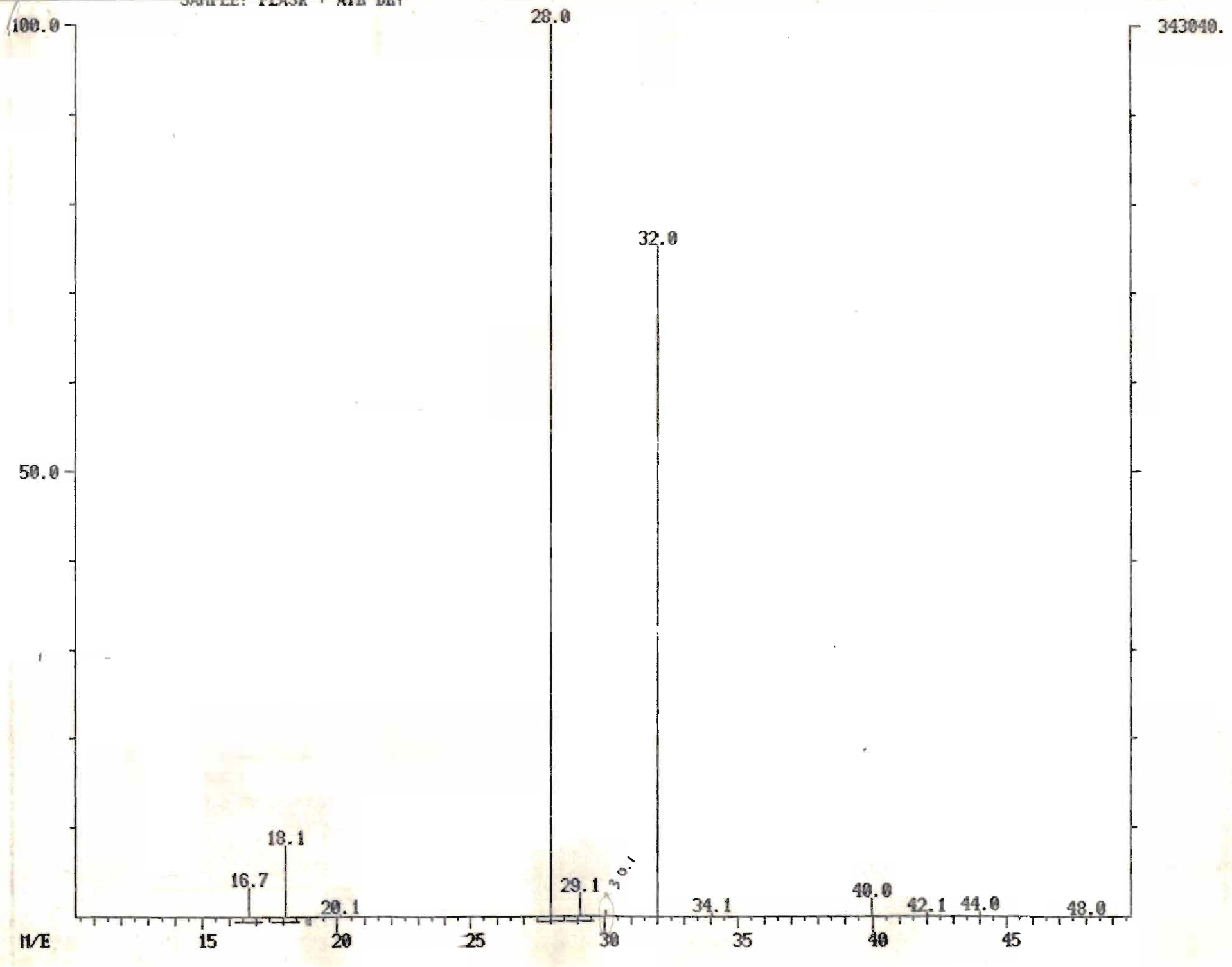
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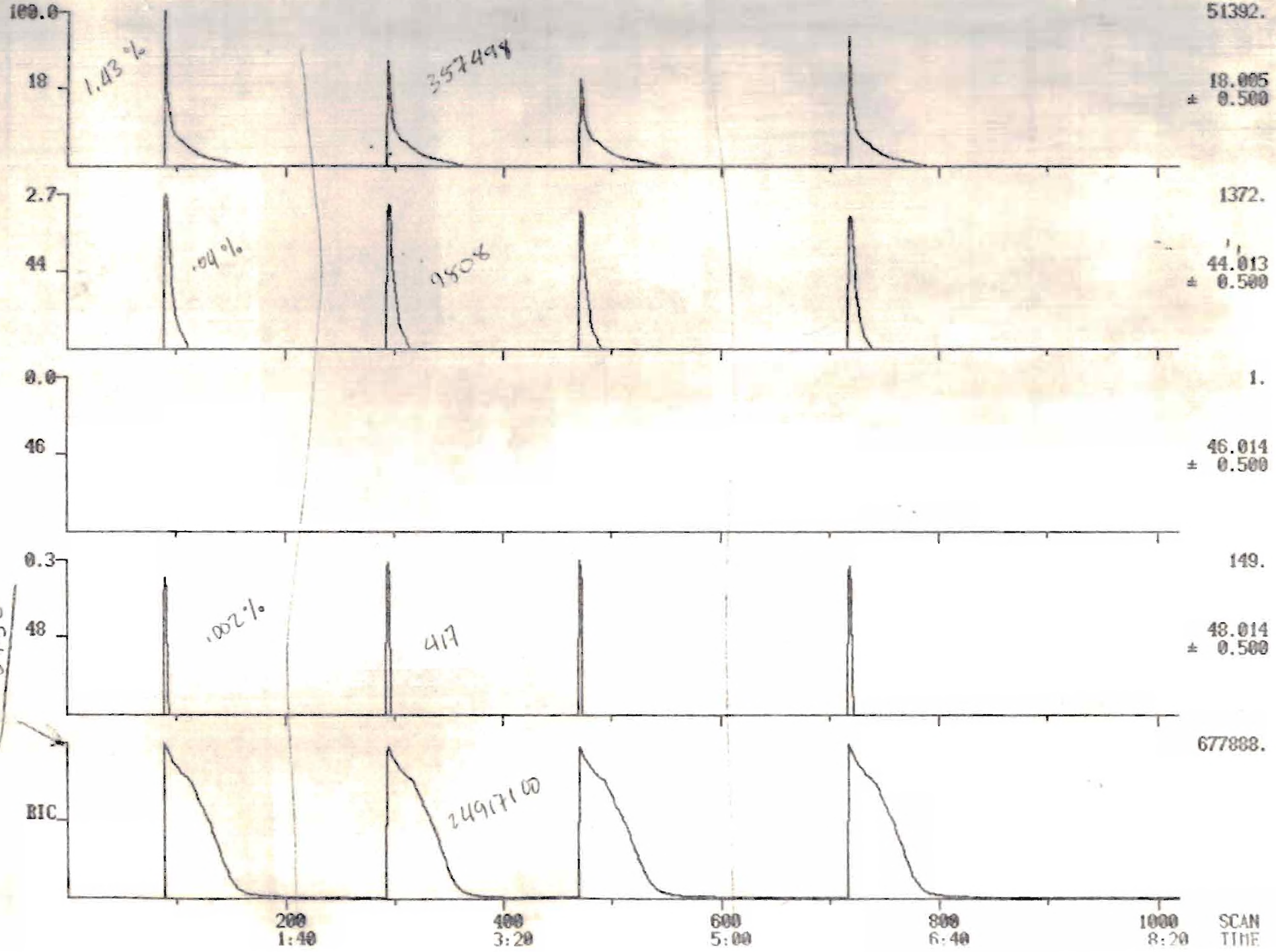
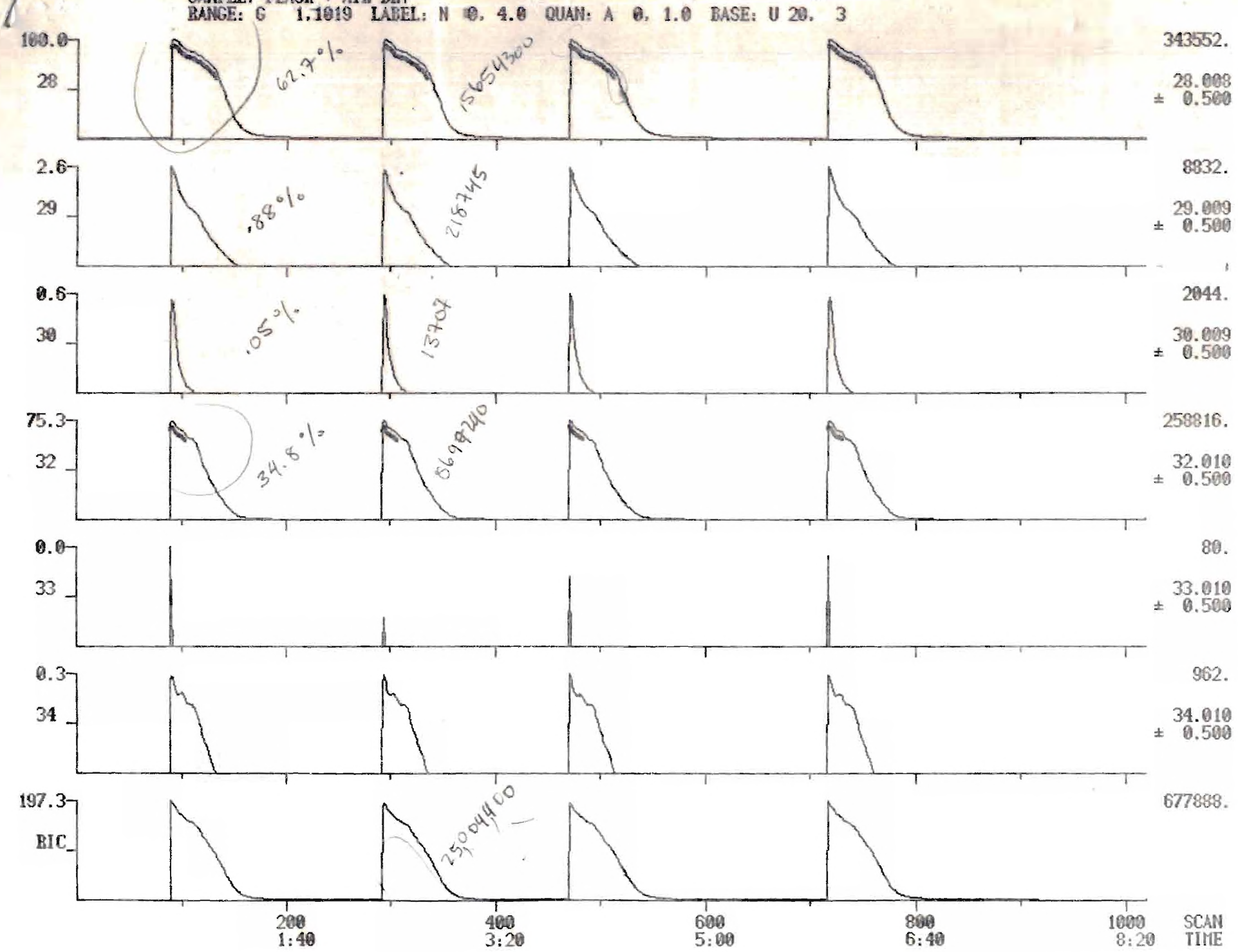
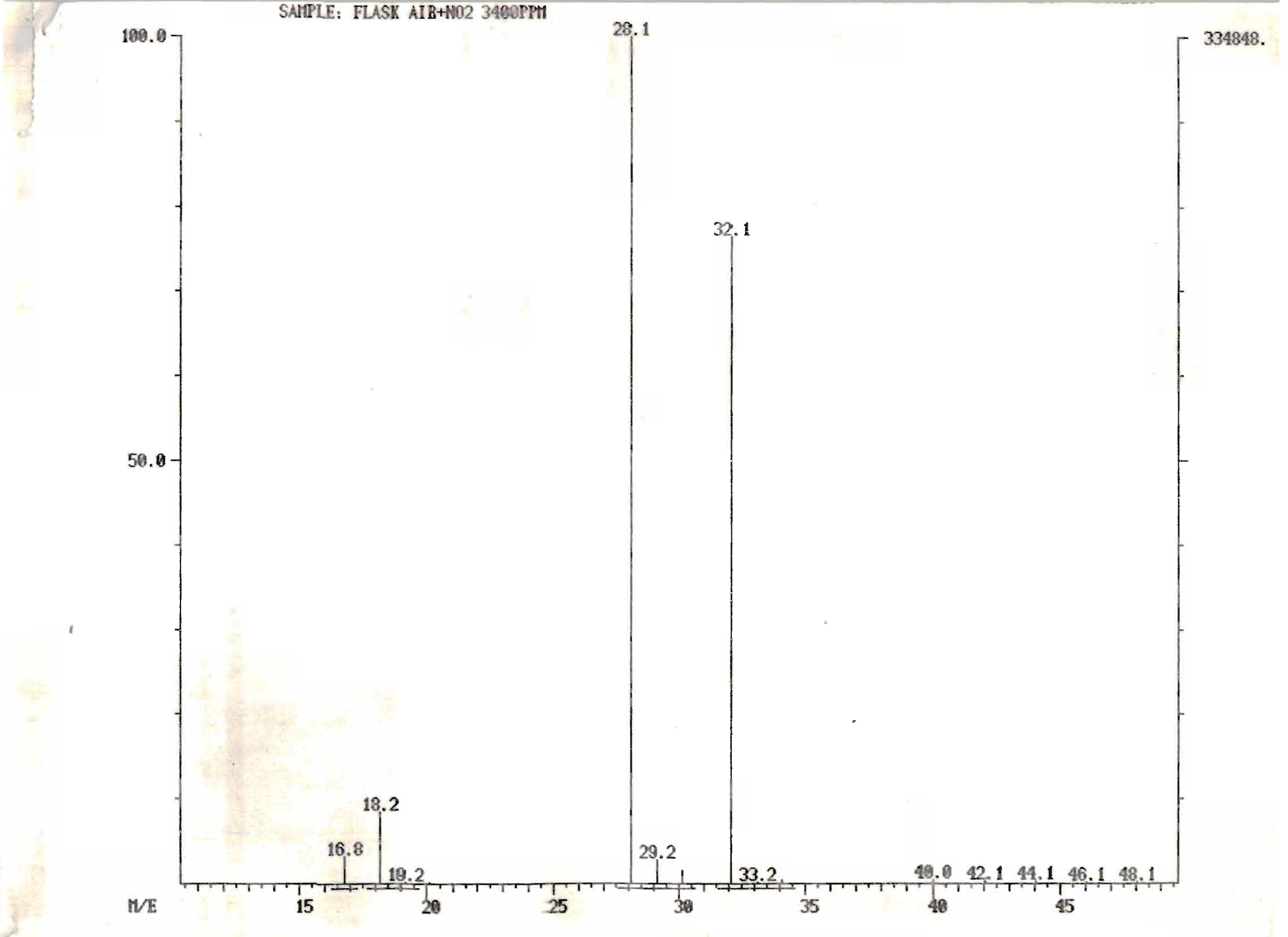
100.0
50.0
343040.
28.0
32.0
18.1
16.7
20.1
29.1
30.1
34.1
40.0
42.1
44.0
48.0
M/E
15
20
25
30
35
40
45

RANGE: G 1.1619 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3




RANGE: G 1.1019 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
100.0
28
62.7%
15654300
343552.
28.008
± 0.500
2.6
29
.88%
218745
8832.
29.009
± 0.500
0.6
30
.05%
13707
2044.
30.009
± 0.500
75.3
32
34.8%
6698240
258816.
32.010
± 0.500
0.0
33
80.
33.010
± 0.500
0.3
34
962.
34.010
± 0.500
197.3
RIC
25004400
677888.
200
1:40
400
3:20
600
5:00
800
6:40
1000
8:20
SCAN
TIME


SAMPLE: FLASK AIR+NO2 3400PPM
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50.0
334848.
28.1
32.1
18.2
16.8
19.2
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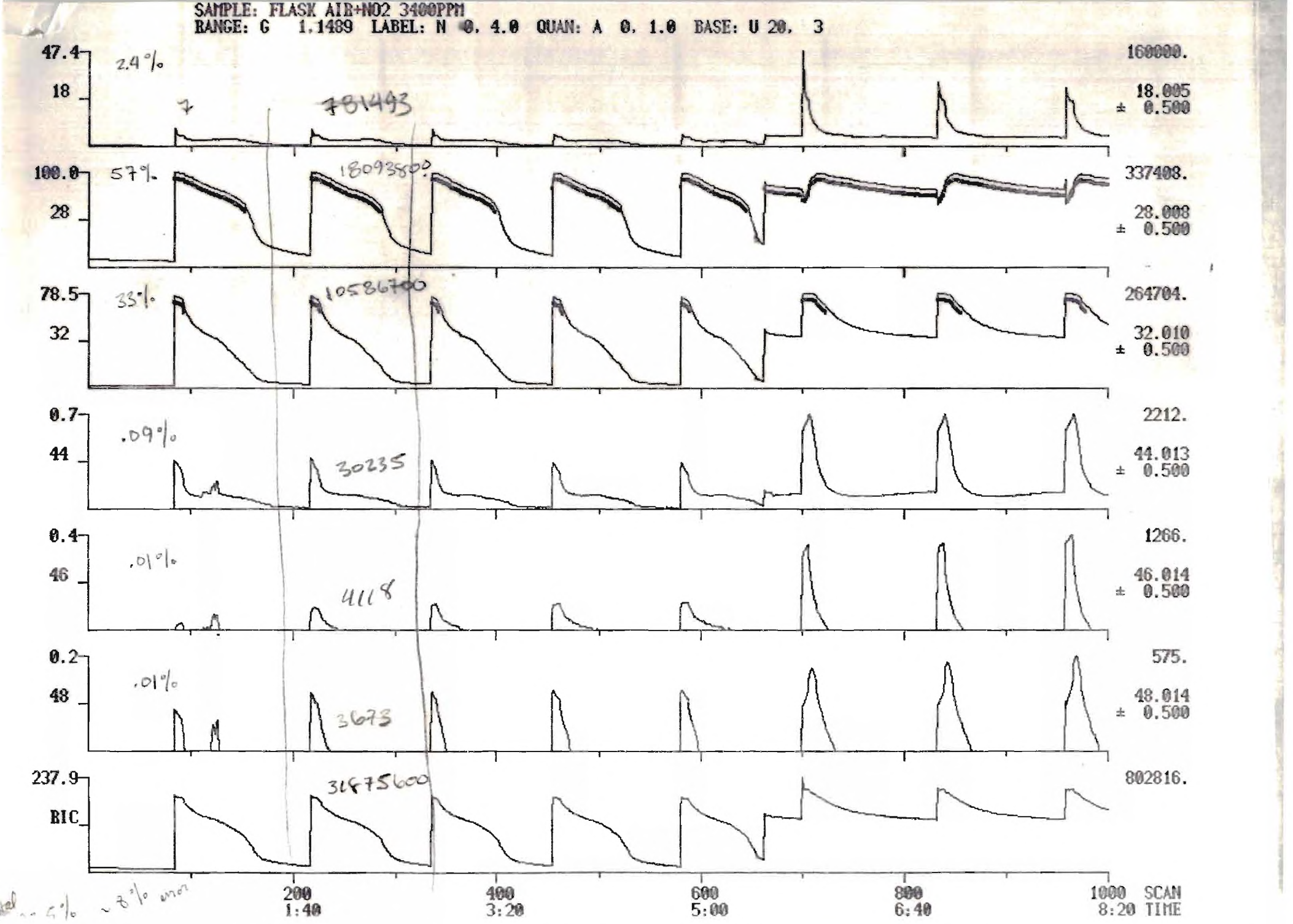
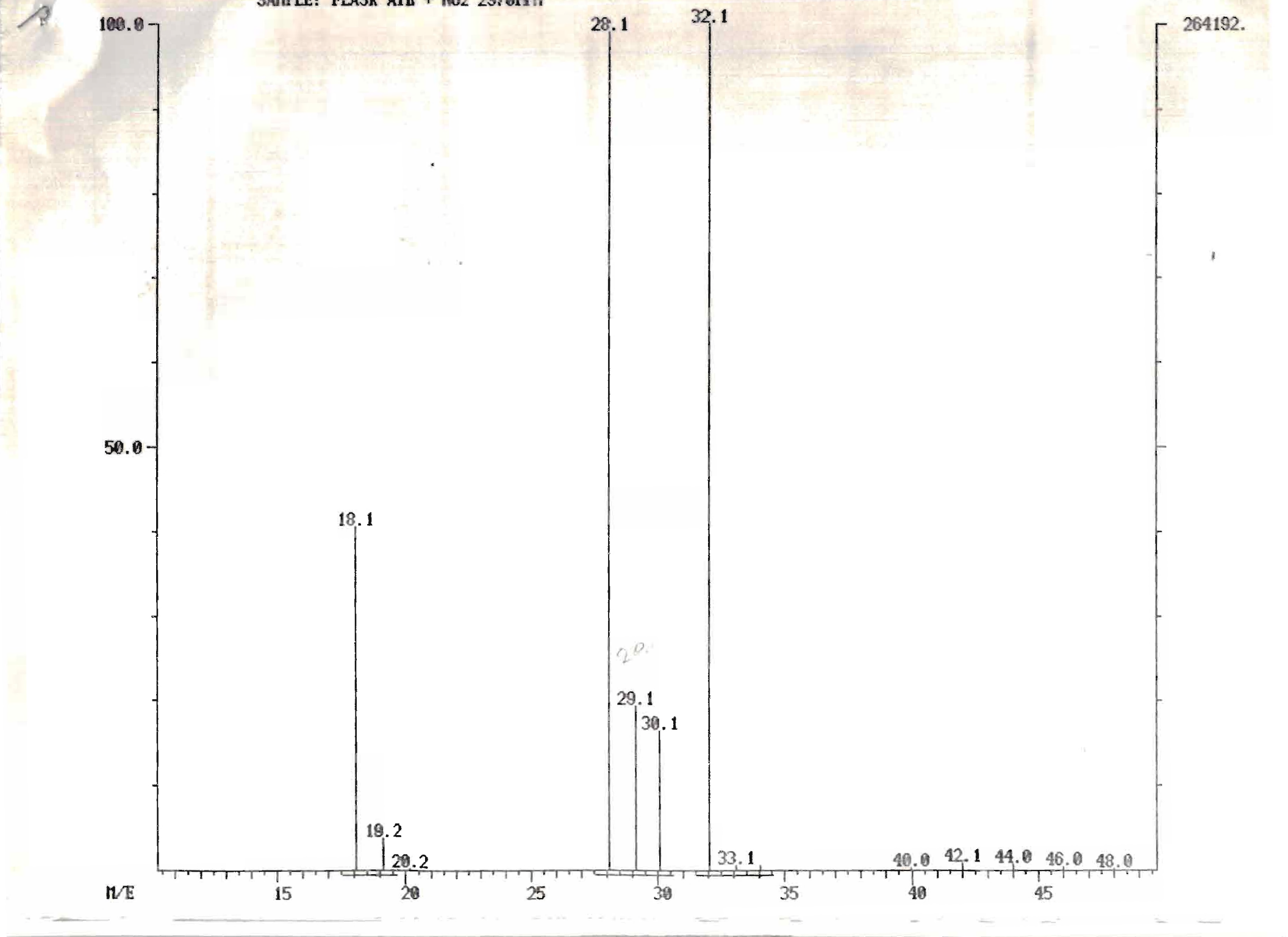
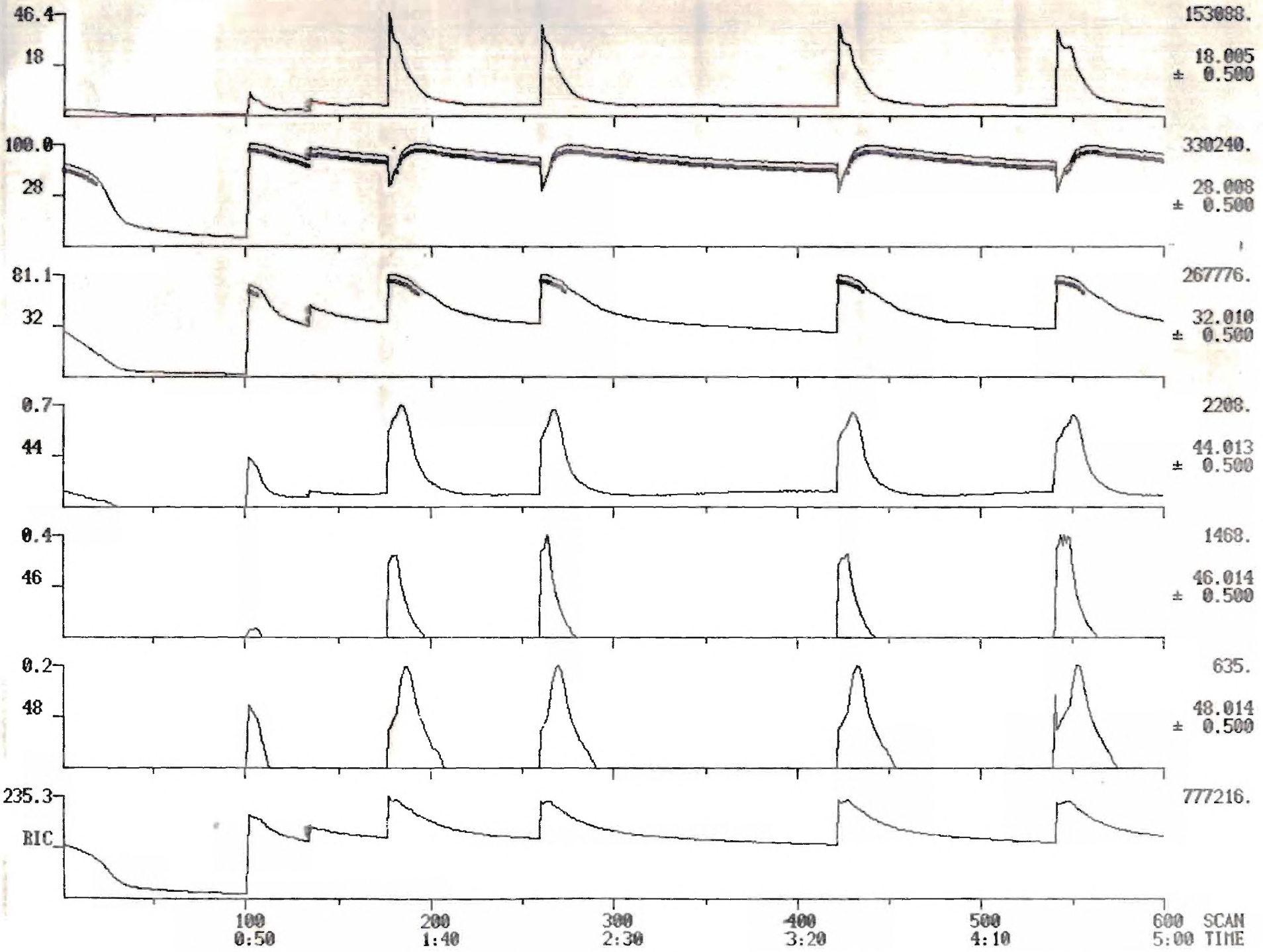
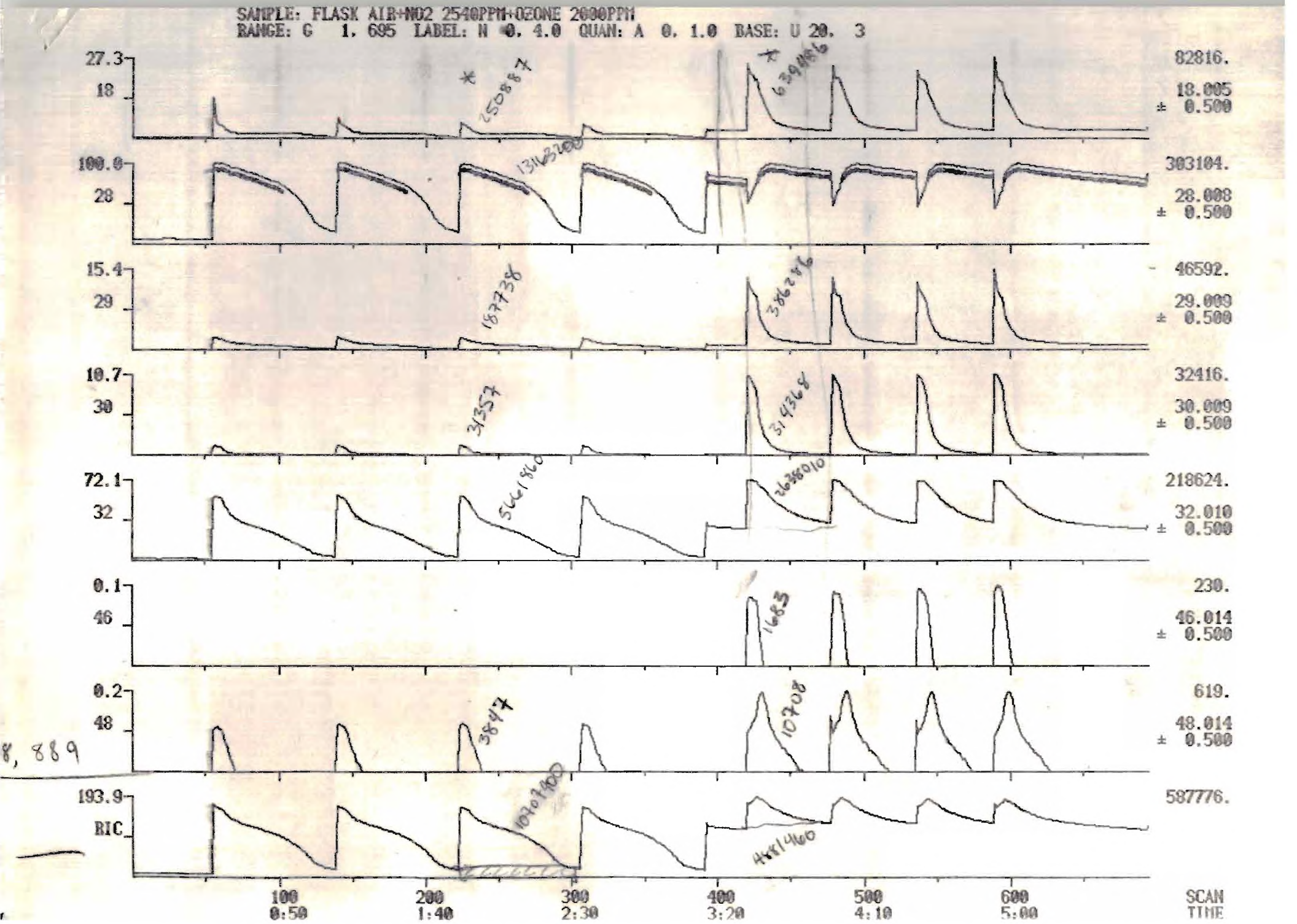
M/E
15
20
25
30
35
40
45

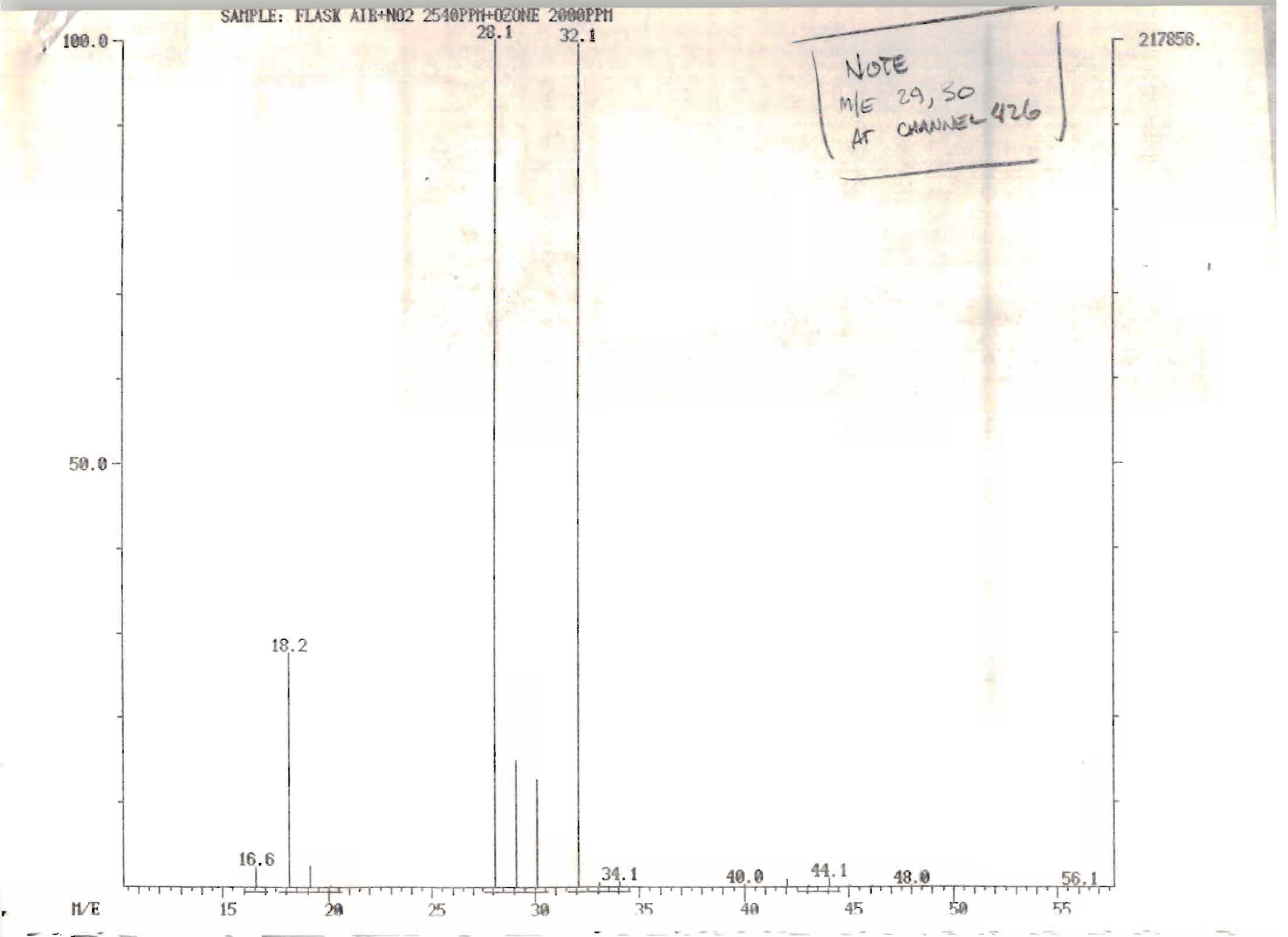

SAMPLE: FLASK AIR+NO2 3400PPM
RANGE: G 1.1489 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
47.4
2.4%
18
781493
160000.
18.005
± 0.500
100.0
57%
28
18093500
337408.
28.008
± 0.500
78.5
33%
32
10586700
264704.
32.010
± 0.500
0.7
.09%
44
30235
2212.
44.013
± 0.500
0.4
.01%
46
4118
1266.
46.014
± 0.500
0.2
.01%
48
3673
575.
48.014
± 0.500
237.9
RIC
31475600
802816.
200
1:40
400
3:20
600
5:00
800
6:40
1000 SCAN
8:20 TIME

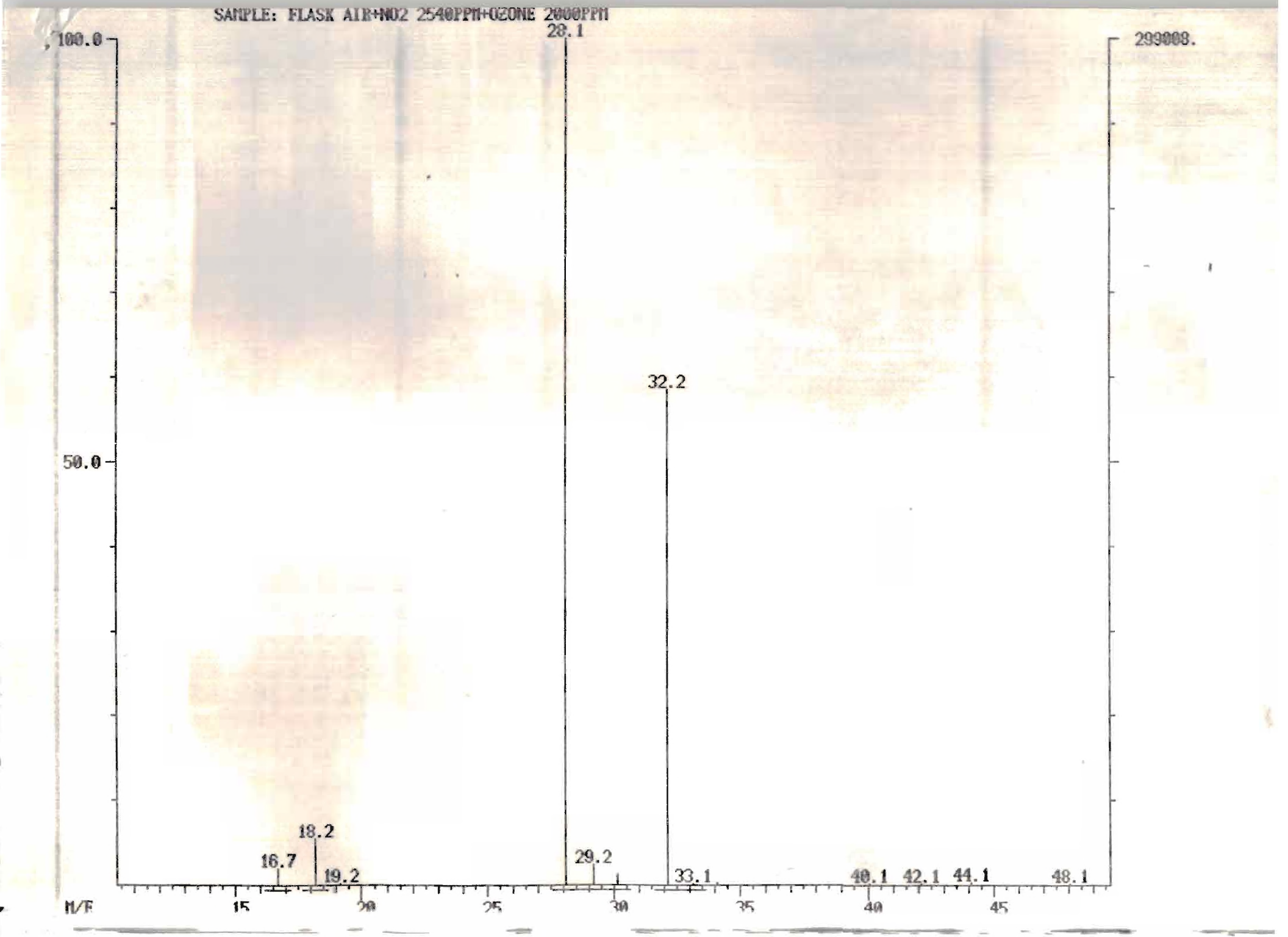

100.0
50.0
264192.
28.1
32.1
18.1
29.1
30.1
19.2
20.2
33.1
40.0
42.1
44.0
46.0
48.0
M/E
15
20
25
30
35
40
45

RANGE: G 1. 641 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3

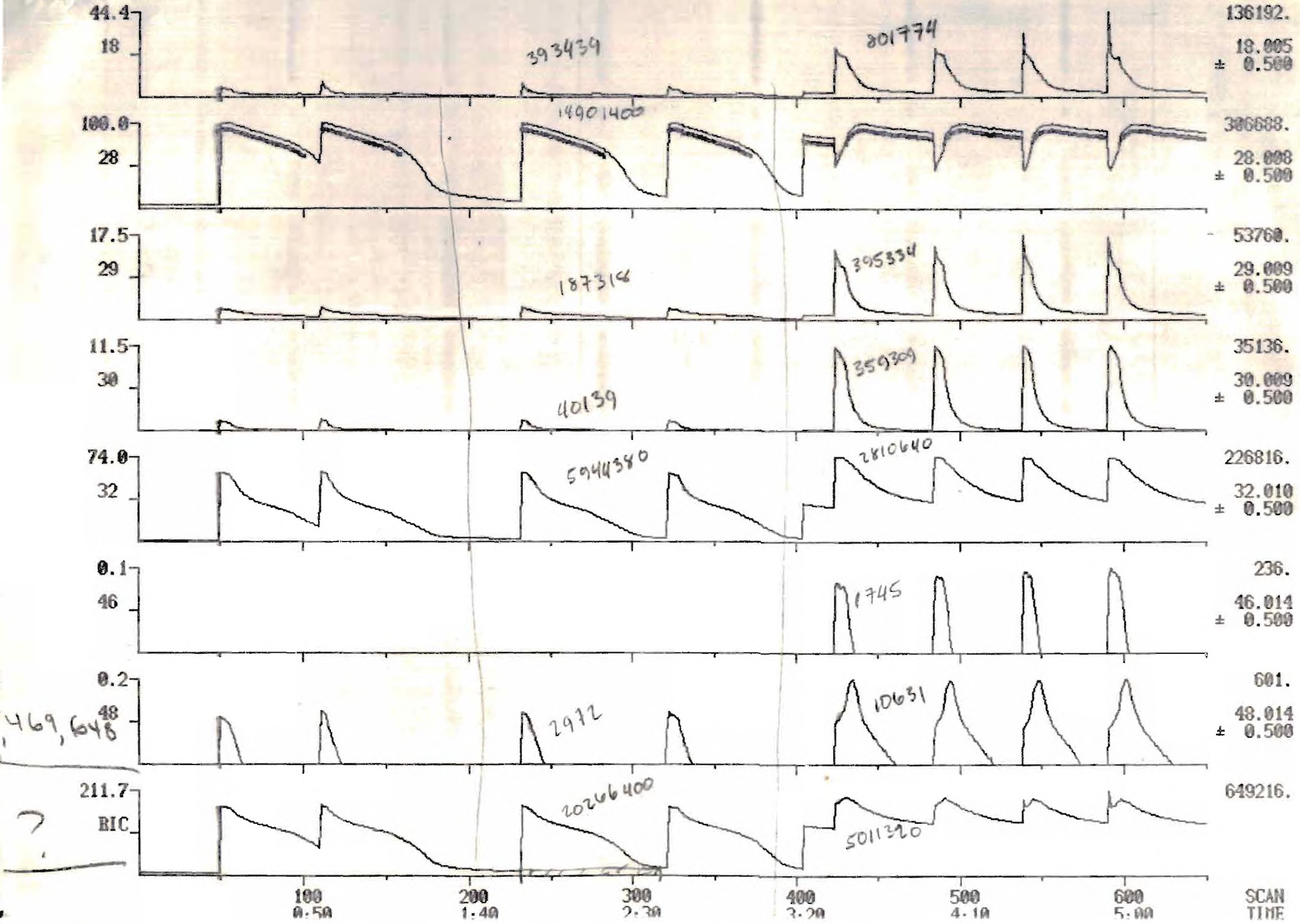


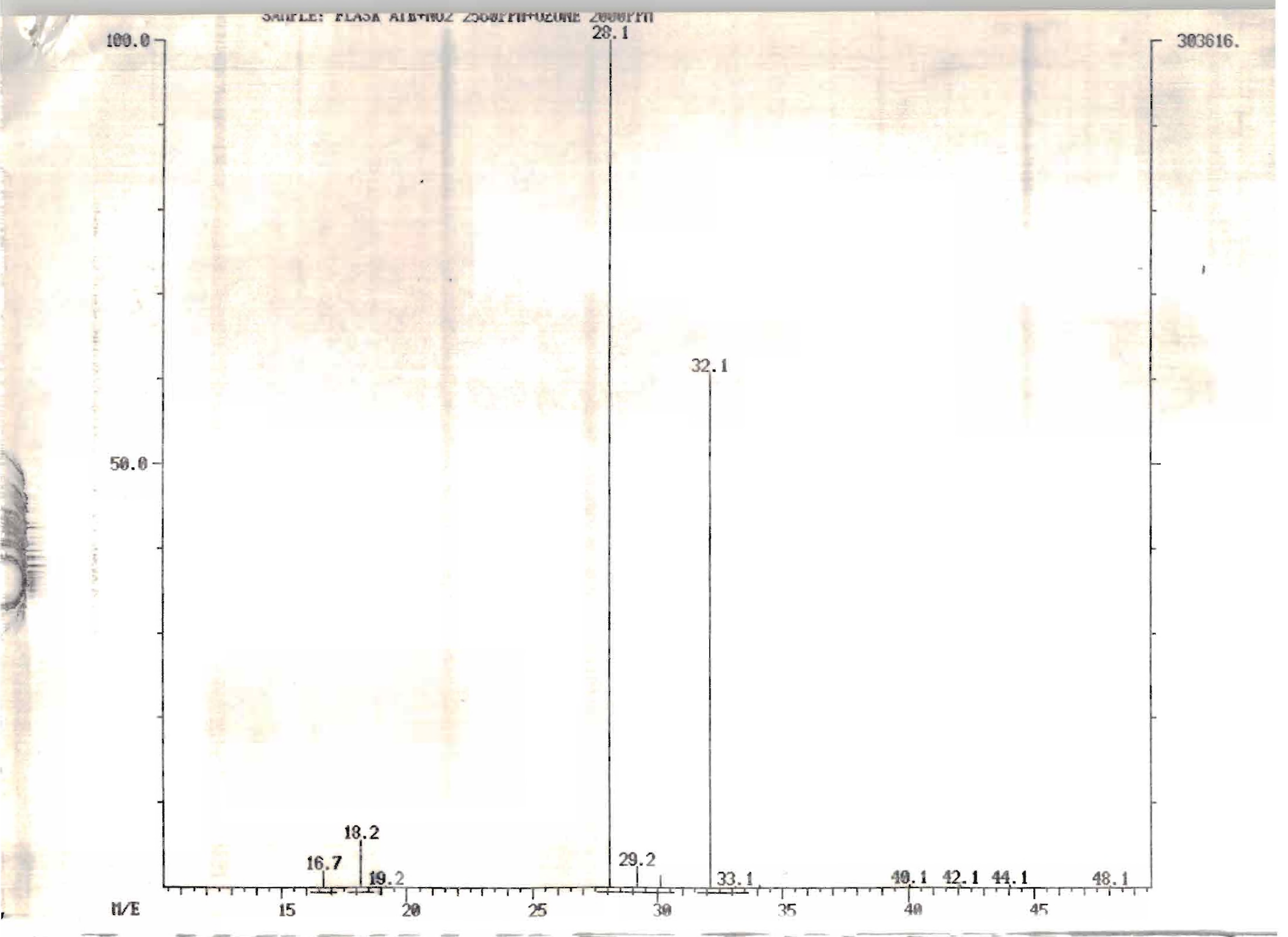

SAMPLE: FLASK AIR+NO2 2540PPM+OZONE 2000PPM
RANGE: G 1. 695 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
27.3
18
82816.
18.005
± 0.500
100.0
28
303104.
28.008
± 0.500
15.4
29
46592.
29.009
± 0.500
10.7
30
32416.
30.009
± 0.500
72.1
32
218624.
32.010
± 0.500
0.1
46
230.
46.014
± 0.500
0.2
48
619.
48.014
± 0.500
193.9
RIC
587776.
100
0:50
200
1:40
300
2:30
400
3:20
500
4:10
600
5:00
SCAN
TIME


SAMPLE: FLASK AIR+NO2 2540PPM+OZONE 2000PPM
100.0
50.0
217856.
28.1
32.1
18.2
16.6
34.1
40.0
44.1
48.0
56.1
M/E
15
20
25
30
35
40
45
50
55
NOTE
M/E 29, 30
AT CHANNEL 426

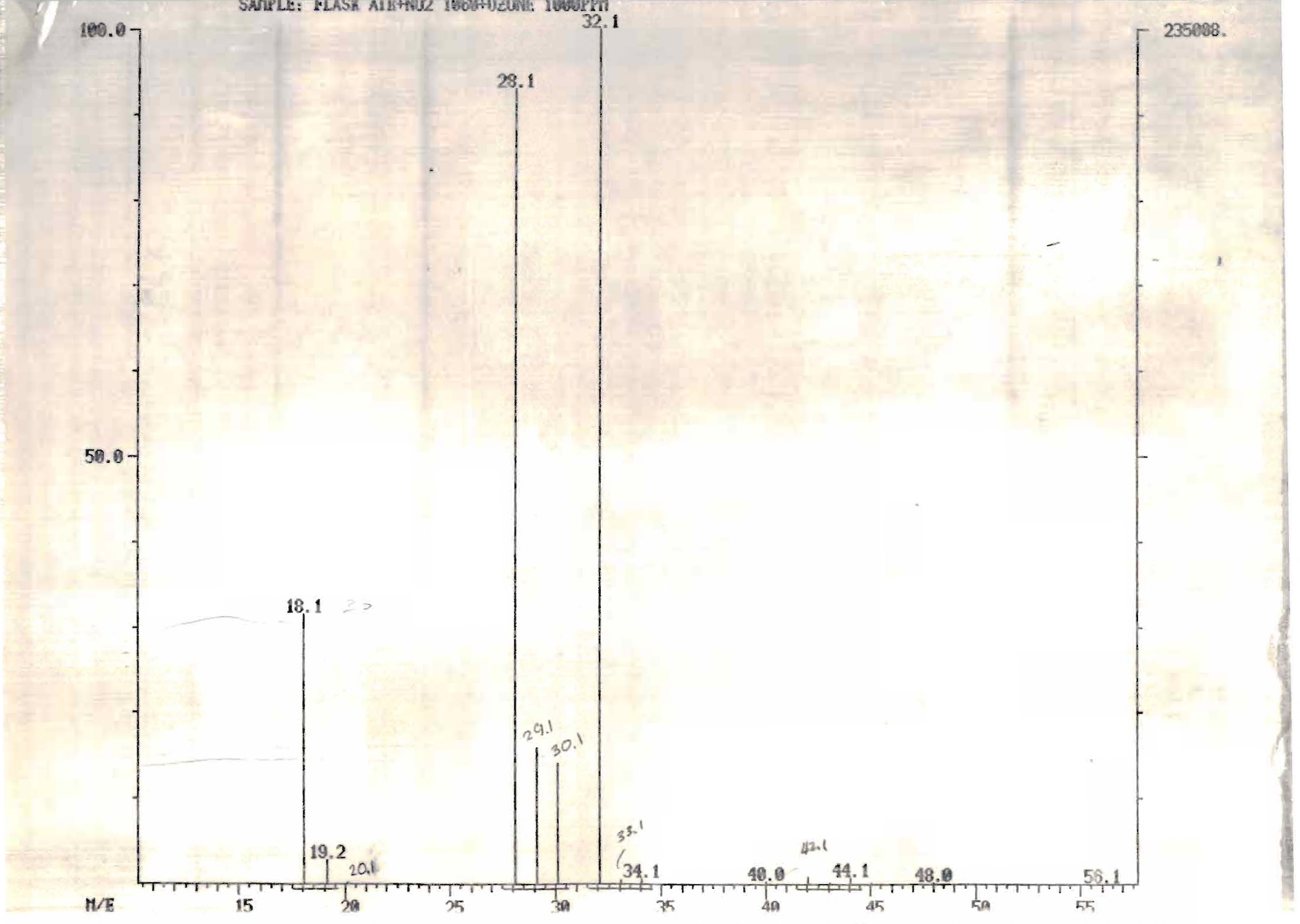

SAMPLE: FLASK AIR+NO2 2540PPM+OZONE 2000PPM
100.0
50.0
28.1
299008.
32.2
18.2
16.7
19.2
29.2
33.1
40.1
42.1
44.1
48.1
M/E
15
20
25
30
35
40
45

SAMPLE: FLASK AIR+NO2 2560PPM+OZONE 2000PPM
RANGE: G 1. 650 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: B 20. 3

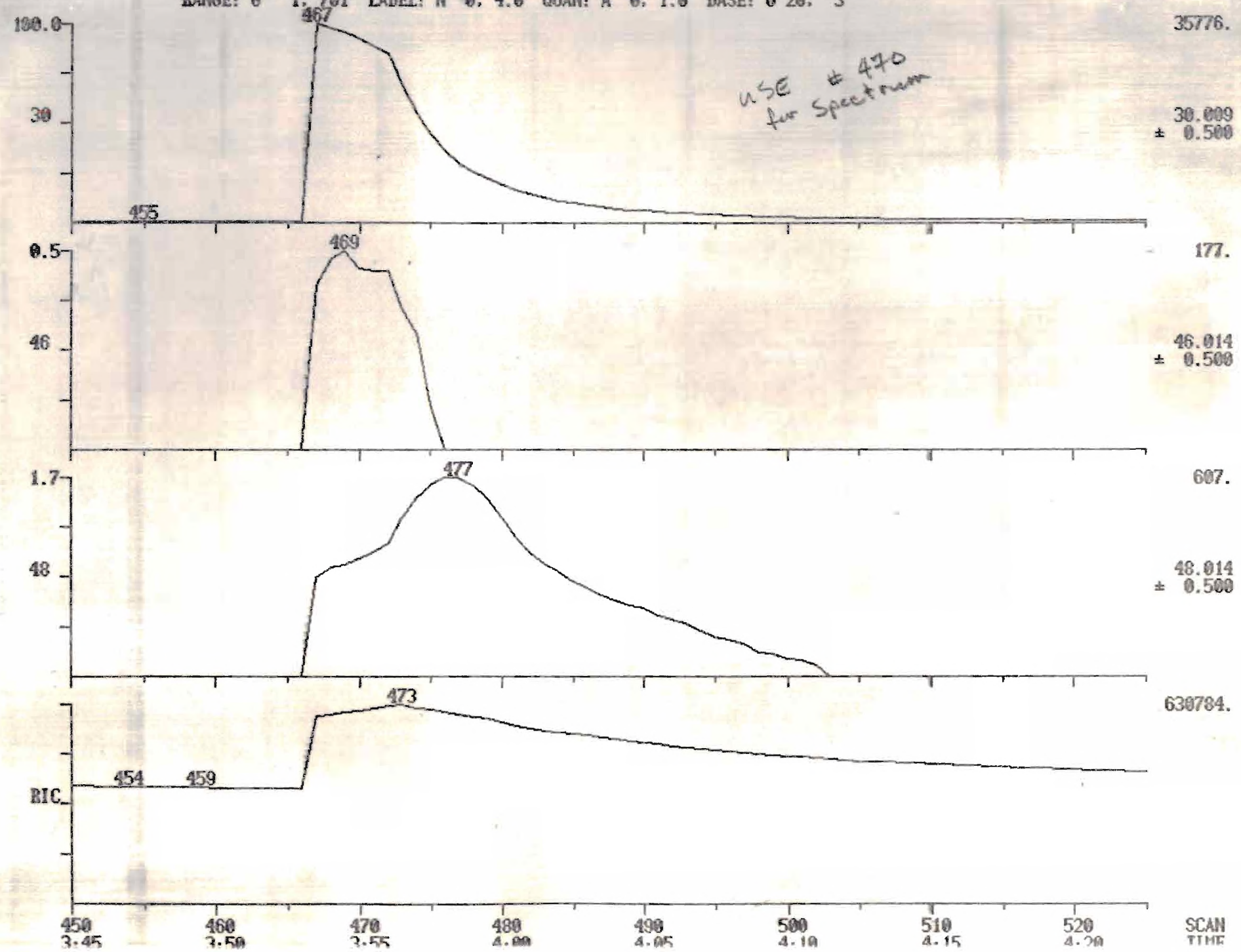



100.0
50.0
303616.
28.1
32.1
18.2
16.7
19.2
29.2
33.1
40.1
42.1
44.1
48.1
M/E
15
20
25
30
35
40
45

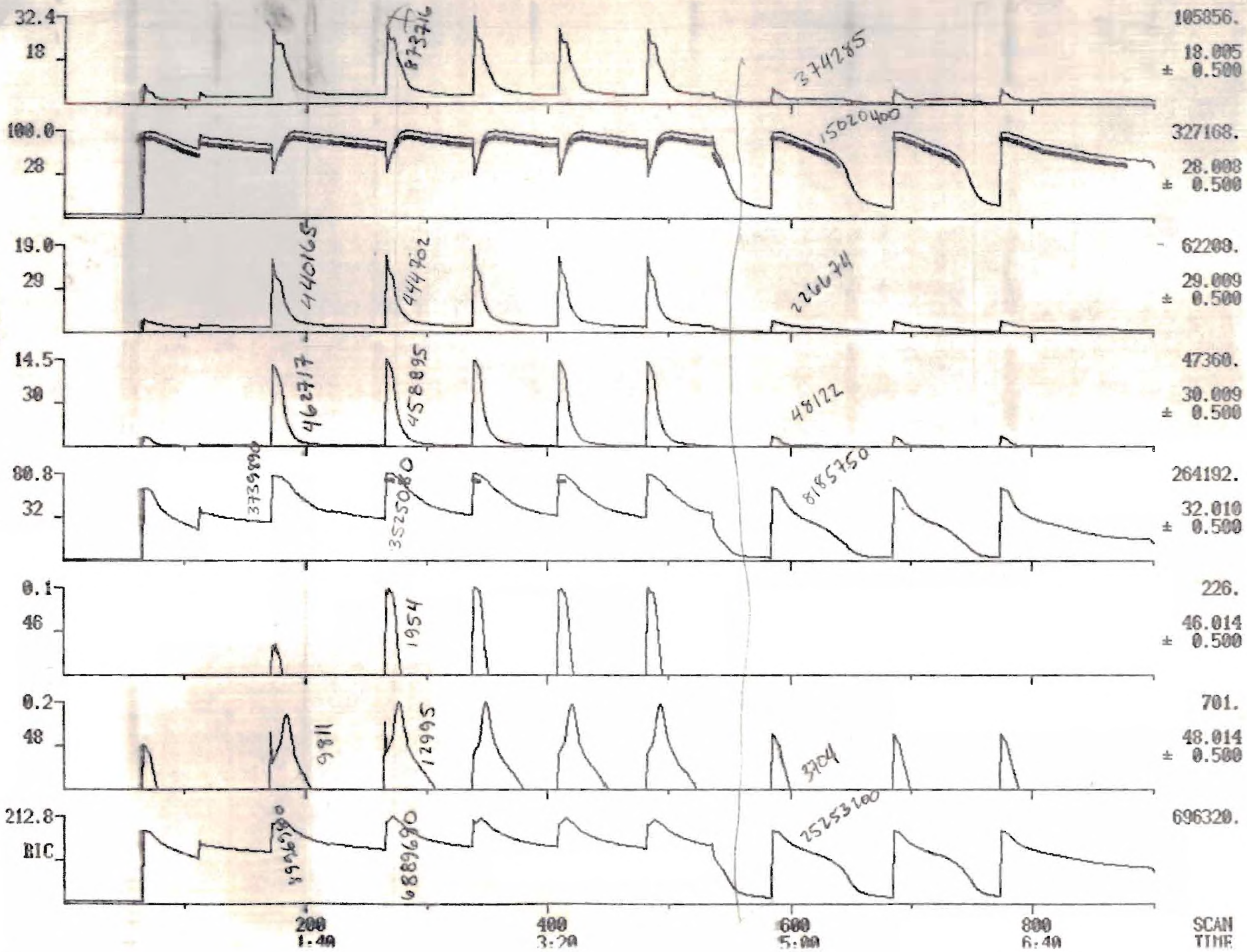
SAMPLE: FLASK AIR+NO2 1000+OZONE 1000PPM
RANGE: G 1. 701 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3

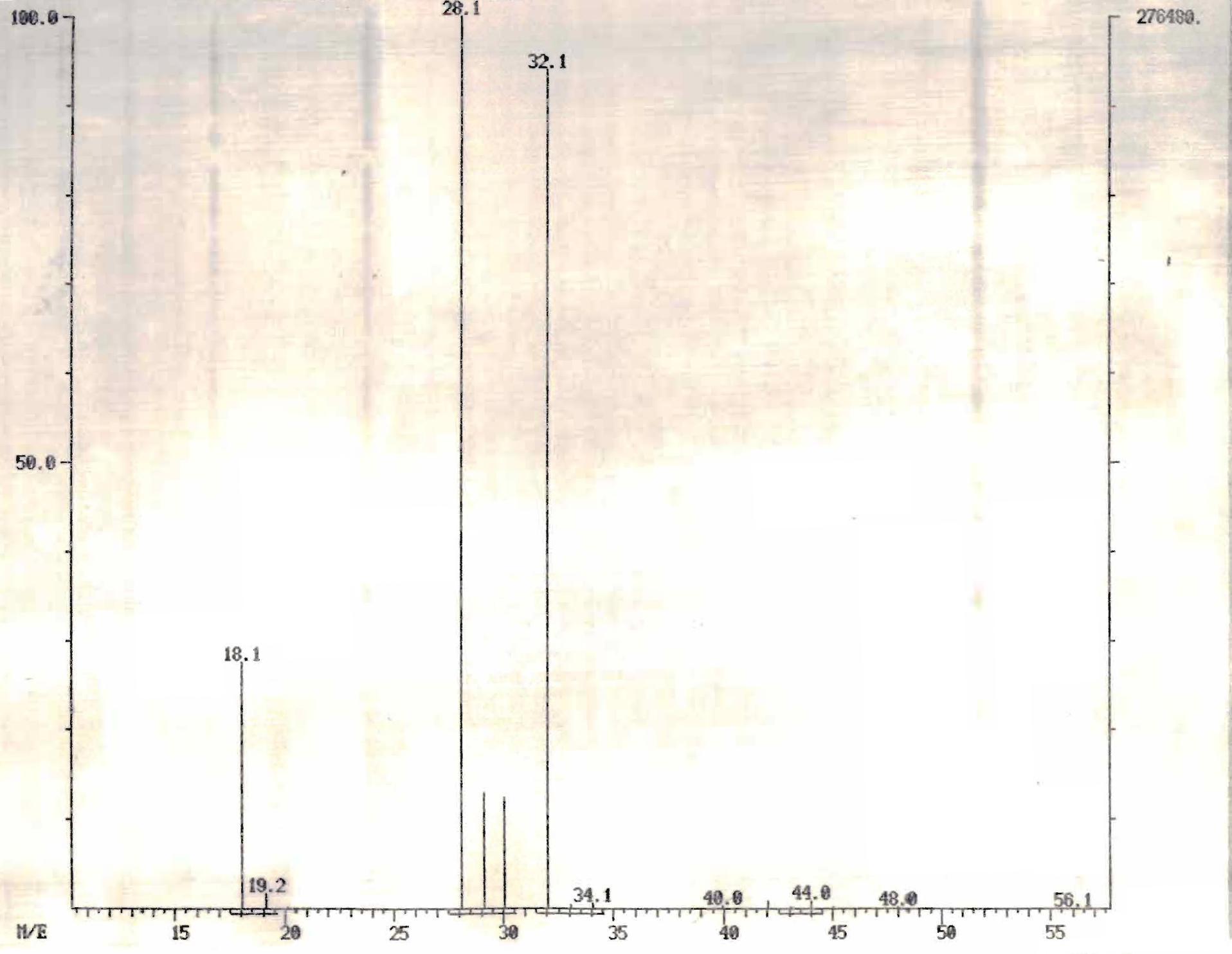

SAMPLE: FLASK A1B+N02 1000+02ONE 1000PPM
100.0
50.0
32.1
28.1
235008.
18.1
29.1
30.1
19.2
20.1
33.1
34.1
42.1
40.0
44.1
48.0
56.1
M/E
15
20
25
30
35
40
45
50
55

SAMPLE: FLASK AIR+NO2 1000+OZONE 1000PPM
RANGE: G 1. 701 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3

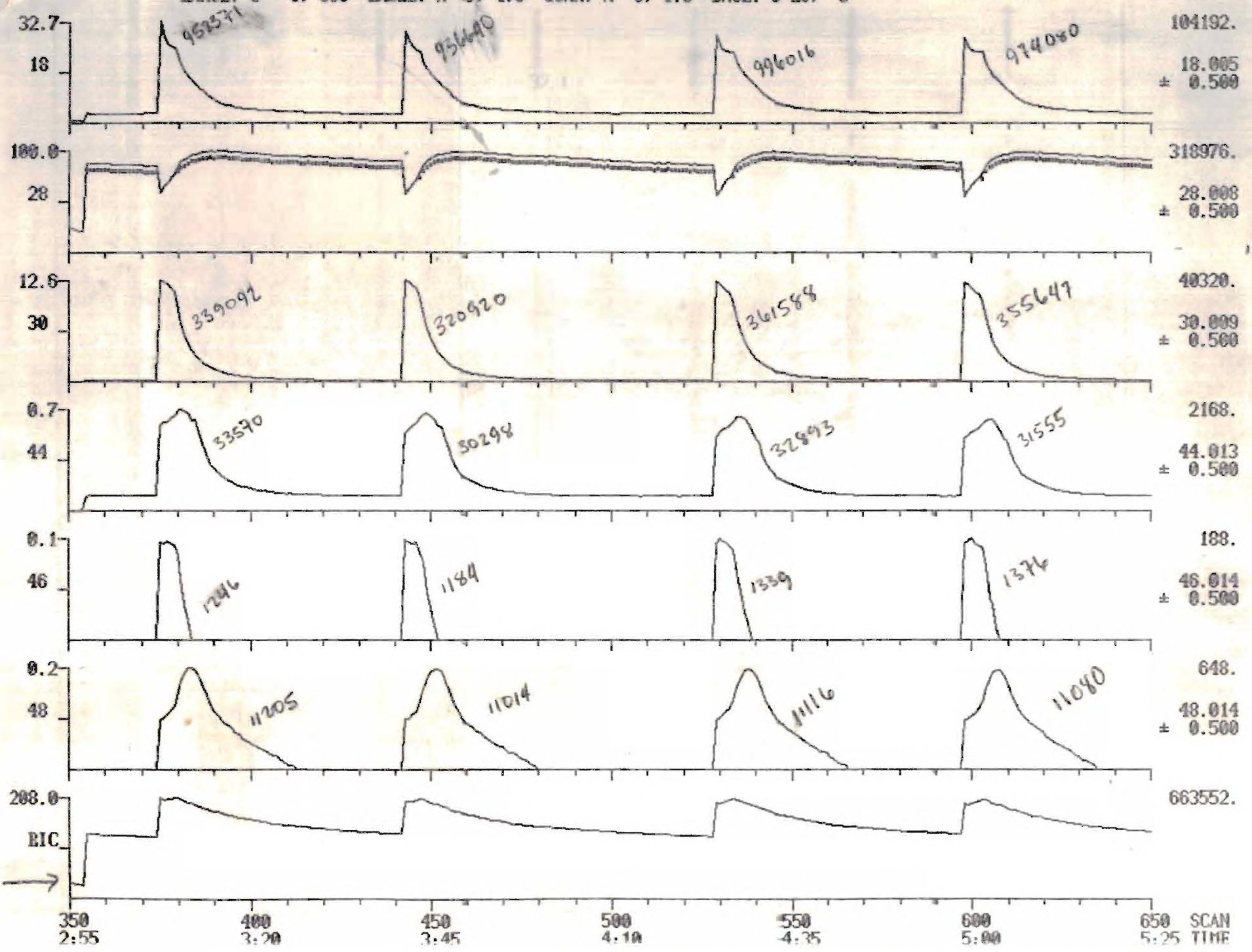


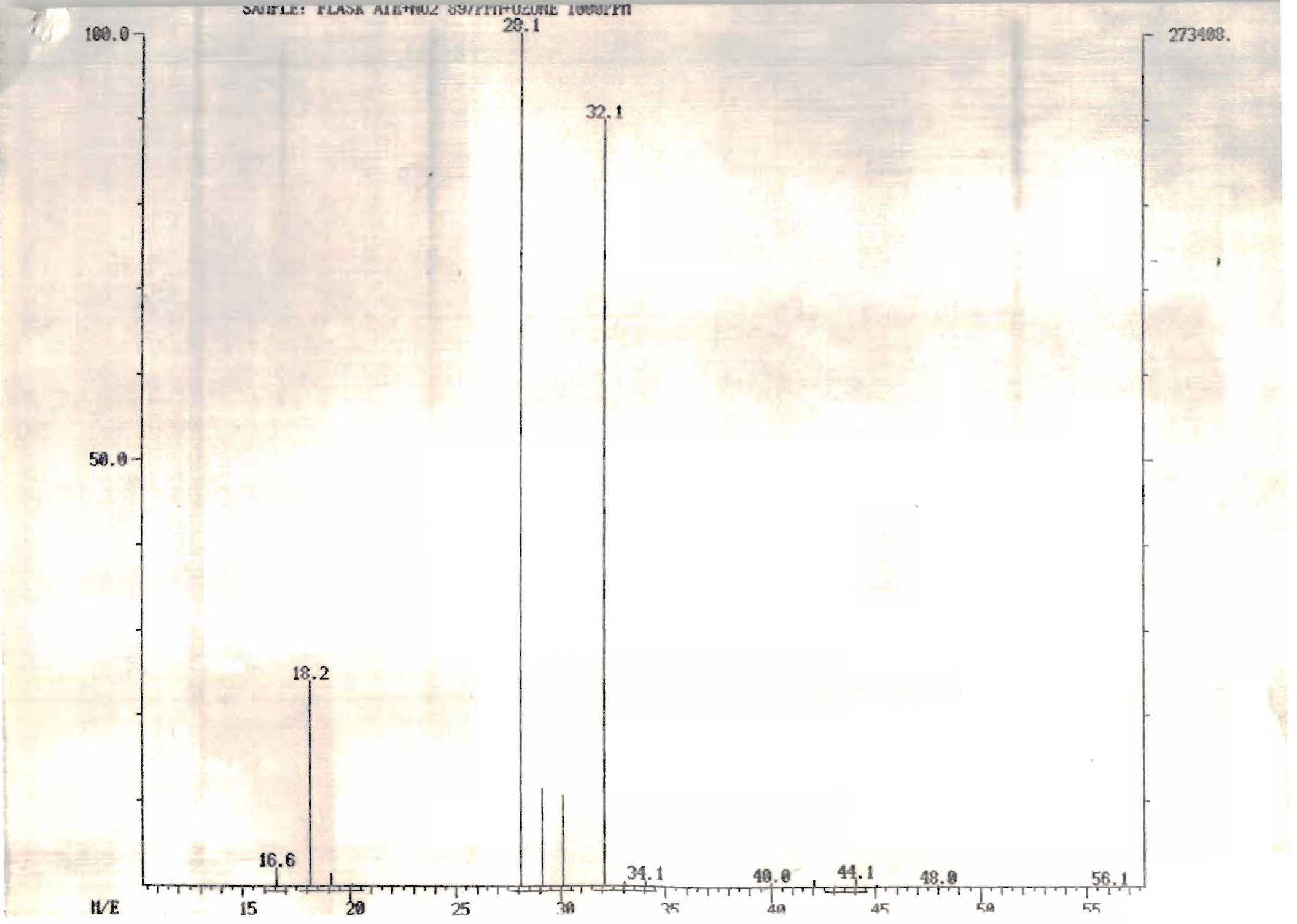
SAMPLE: FLASK AIR+OZONE 1023+002 1590
RANGE: G 1. 925 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: H 20. 3




100.0
276480.
28.1
32.1
50.0
18.1
19.2
34.1
40.0
44.0
48.0
56.1
M/E
15
20
25
30
35
40
45
50
55

SAMPLE: FLASK AIR+NO2 897PPM+OZONE 1000PPM
RANGE: G 1. 689 LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3




100.0
50.0
273408.
28.1
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18.2
16.6
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44.1
48.0
56.1
M/E
15
20
25
30
35
40
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50
55

A-60-602

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

November 9, 1984

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the month of October 1984 on the project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

I. Irradiation

As you witnessed on your recent visit to Georgia Tech, the irradiation process has finally begun. Results from analyses on zeolites with lowest dose (1×10^5 rads) should be out soon.

II. Adsorption and Crystallographic Analysis of Unirradiated Samples

Dr. Tudor Thomas, who is responsible for the adsorption and crystallographic characterization on the project, has given the appended report (also given to you during your recent visit). By way of introduction, Dr. Thomas has spent all of his professional life (35 years) in mordenite materials. He is extremely knowledgeable and one of the foremost experts in this area.

As stated in Dr. Thomas' report for the oxygen adsorption characterization, 3 samples were run: (1) your sample of hydrogen mordenite, (2) your sample of silver mordenite, and (3) a commercial sample of hydrogen mordenite (obtained from Union Carbide). This sample contains a 20% clay binder. The results show that your sample and that of Union Carbide have similar adsorption characteristics (22 wt.%). Dr. Thomas says this value is what is expected. The adsorption of oxygen on silver mordenite, however, is lower than expected and this result indicates that something is unusual with the silver mordenite.

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Crystallographic diffraction patterns were also obtained for 3 samples: your two samples and a third hydrogen mordenite sample that is 100% crystalline. This hydrogen mordenite sample is different from the one used above. This one is 100% crystalline compared to 80% for the other.

The results show that your H-mordenite sample is 79% crystalline and is similar to the Union Carbide commercial sample with 20% clay binder. The story on your silver mordenite sample however is different; it is only 16% crystalline. Dr. Thomas believes that this sample contains a significant amount of elemental silver.

Another conclusion of Dr. Thomas is that the silver exchanged H-mordenite pellets are weak and easily crushed. This comment agrees with the hardness and crushness results reported to you in the August 16 and October 2, 1984 reports.

Since you also raised some questions about the hardness and crushness results, I have discussed these issues with Dr. Benzel from Ceramic Engineering. His comments follow.

Crushness Test: Right cylindrical zeolite pellet is placed between two flat plates and the load is increased until the pellet is crushed. If the cylindrical pellet has smooth and even surface, a line contact between pellet and flat plate is established. If pellet is not smooth and not even, a point contact is established between flat plates and pellet. The load needed to start the crushing process in both cases would be different. This is one reason for the large variability in the results. Another reason is the normal statistical variability of the sample. This variability, according to Dr. Benzel should be about 20%, i.e., standard deviation of only 20%. Your samples exhibit a much larger variation.

Hardness Test: In this test one attempts to measure the load needed to penetrate the pellet surface. The apparatus here is a diamond pyramid indenter which presses on the pellet at a point rather than at a line. The purpose of this test is that if the packing in a column of zeolite is such that contacts among pellets occur at points, rather than lines, then it is useful to know what loads are needed to affect penetrations.

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Dr. Benzel concluded that the silver mordenite is very soft and suggested that if it is desirable to use this material it should be packed in a horizontal rather than in a vertical column.

If you have any questions please let me know.

With best wishes.

Sincerely,

Ratib A. Karam
Interim Director
Nuclear Research Center

pc: Dr. J. Benzel
Dr. T. Thomas

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

Zeolite Research Program

4 October 1984

TO: Ratib A. Karam
Nuclear Engineering

FROM: T. L. Thomas

SUBJECT: Savannah River Contract (A60-602) ← Project number

We have received from you to this date two samples for evaluation, H-mordenite and silver exchanged H-mordenite, both were as received by you from your sponsor.

The evaluation results are as follows:

1. Oxygen adsorption of 75 torr and -196°C after activation at 300°C under vacuum:

H-mordenite sample	22.7 wt - %
Ag - mordenite sample	9.2 wt - %
commercial H-mordenite	22.6 wt - %

2. X-ray diffraction patterns were obtained for each of the 2 samples and for a sample of commercial H-mordenite powder with 100% crystallinity. The % crystallinity of the samples relative to 100% cyrstalline material was:

H-mordenite sample	79. %
Ag-mordenite sample	16. %

The Ag-mordenite X-ray pattern had several new peaks which have not been identified.

The following conclusions can be made from these data:

1. The H-mordenite pelleted sample has normal oxygen adsorption and crystallinity when compared to commercial pelleted H-mordenite material, which contains 80% powder and 20% clay binder.

2. The silver-exchanged H-mordenite pelleted material has only 20% of the crystallinity of H-mordenite pelleted material and only 40% of the adsorption capacity of H-mordenite pellets. Depending on the service intended, this material could have only marginal utility.

It should be noted that the H-mordenite pellets as received, have fair hardness that should be acceptable; however, the silver exchanged H-mordenite pellets are very weak and are easily crushed, such that they would be unacceptable in most service.

A-60-602

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DESIGNING TOMORROW TODAY

December 21, 1984

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the month of November, 1984 on the project entitled "Characterization of Changes in Adsorbent Properties Under High Radiation Doses."

I. Gas Analysis

As you agreed to by telephone and as reported to you in the progress report for the months of August and September, 1984, Dr. Douglas Davis of Georgia Tech has analyzed the mixtures of gas containing 1000 ppm NO_2, 1000 ppm O_3 in dry and saturated air. The analysis of the initial preparation took place 5-6 hours after the gas was mixed. It was not possible to do the analysis sooner than 5-6 hours because of the physical separation between the laboratories where the gas was prepared and mixed and where it was analyzed. The gas was analyzed for NO, NO_2 and O_3. The results are:

	NO	NO_2	O_3
Dry gas* ppb	3000	121	500
Saturated gas** ppb	198	171	700

* Gas mixture content based on partial pressure makeup of standard dry air from Matheson + 923 ppm NO_2 and 1001 ppm NO_3.

** Gas mixture content based on partial pressure makeup of standard air from Matheson that was saturated with H_2O + 1026 ppm NO_2 and 1001 ppm O_3.

Dr. Davis states that the uncertainty associated with these numbers is $\pm$ 15%. He also said that he was not surprised to find such a low concentration of NO_2, NO, and O_3 in the initial gas mix because the components react together quickly to form HNO_3, NO_3, N_2O_5 and others.

Dr. Davis also analyzed the gas from 8 sample holders which were irradiated to a total dose of 1 x 10^8 rads. The results are given in Table I. It is not apparent that a dose of 10^8 rads alters the gas mixture in the sample holders.

II. Density Measurements

The average density for the first 8 irradiated samples and two control samples are given in Table II. Note that the control (unirradiated) samples gave the same value as the irradiated samples and that both values are slightly higher than previous values sent to you without inclusion of the gas mixture (see progress report for months of August and September). It is not known at this point whether or not the difference is due to systematic error or statical error. In these measurements the statistical error is larger because of the limited number of zeolite pellets in each sample holder. Dr. Benzel has not yet evaluated the statistical error.

III. Crushness and Hardness Tests

These tests have <u>not</u> been performed yet because Dr. Benzel was extremely busy during November and early December, 1984.

IV. Adsorption and Crystallographic Properties of Irradiated Samples

Dr. Tudor Thomas performed the crystallographic and adsorption characterization on eight irradiated samples, 2 control samples and two additional samples supplied by you on my recent visit to SRL. The results are given in Table III.

It appears that:

1. The H-mordenite materials are relatively unaffected by the addition of ozone and NO_2 and exposure to radiation. The retention of crystallinity and oxygen capacity is indicated.

2. Ag-mordenite is much less stable than the H-mordenite to these conditions.

Sincerely,

Ratib A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Attachments

TABLE I

Gas Analysis for NO and NO_2 from Sample Holders

Irradiated to 1 x 10^8 Rads

Sample Holder #	Contents	Radiation Dose (Rads)	NO PPB[a]	NO_2 PPB
5	Dry Ag Zeolite + Gas	1.E + 8	136	$\leq$935[b]
6	Dry Ag Zeolite + Gas	1.E + 8	30	511
7	Sat Ag Zeolite + Gas	1.E + 8	23	216
8	Sat Ag Zeolite + Gas	1.E + 8	16	120
9	Dry H Zeolite + Gas	1.E + 8	37	491
10	Dry H Zeolite + Gas	1.E + 8	Used for std. addition[c]	
11	Sat H Zeolite + Gas	1.E + 8	19	236
12	Sat H Zeolite + Gas	1.E + 8	21	264
41	Dry Ag Zeolite + Gas	Not Irradiated[d]	21	216
43	Sat Ag Zeolite + Gas	Not Irradiated	26	122

[a] No measurements uncertainty is $\pm$15%; NO_2 is $\pm$20%

[b] This sample was somehow contaminated with the NO_2 Standard according to Dr. Davis.

[c] This sample was used to add to it a known NO and NO_2 amounts. The addition was 100 ppm of each. The measurement showed that only the added parts were detected indicating that the amounts of NO and NO_2 initially present were small relative to 100 ppm's. The other measurements confirm this.

[d] Sample holders Nos. 41 and 43 were control samples filled with the same gas at the same time as the others but were not irradiated.

TABLE II
Zeolite Samples (First Set)

SAMPLE	TYPE	RADS	GAS	DENSITY (gm/cm^3)
5	Ag	10^8	Dry	2.444
6	Ag	10^8	Dry	2.444
7	Ag	10^8	Saturated	2.443
8	Ag	10^8	Saturated	2.431
9	H_2	10^8	Dry	1.996
10	H_2	10^8	Dry	2.019
11	H_2	10^8	Saturated	1.974
12	H_2	10^8	Saturated	1.973
41	Ag	0	Dry	2.443
43	Ag	0	Saturated	2.436

TABLE III
Adsorption and Crystallographic Properties

Sample Holder #	Contents	Irradiation Dose	% Crystallinity[a,b]	Capacity-wt%[c]
5	dry Ag-M	1×10^8 Rads	19.	10.1
6	dry Ag-M	1×10^8 Rads	16.	8.5
7	sat Ag-M	1×10^8 Rads	16.	10.4
8	sat Ag-M	1×10^8 Rads	18.	9.7
9	dry H-M	1×10^8 Rads	83.	22.6
10	dry H-M	1×10^8 Rads	91.	22.4
11	sat H-M	1×10^8 Rads	89.	22.2
12	sat H-M	1×10^8 Rads	70.	21.6
41	dry Ag-M	0.0	16.	10.8
43	sat Ag-M	0.0	15.	12.3
	commercial H-mordenite	0.0	100.	22.8
	Norton 900-H	0.0	100.	22.9
	Ag-mordenite SAI-1980	0.0	26.	19.5

[a] X-ray diffraction pattern of the sample relative to a standard H-mordenite sample of 100% crystallinity

[b] All results compare to a commercial H-mordenite powder with 100% crystallinity.

[c] Oxygen adsorption capacity at 75 torr and $-196^{\circ}C$ after activation at $300^{\circ}C$ under vacuum

A-60-602

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

January 23, 1985

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the month of December, 1984 on the project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

I. Gas Analysis

The one billion rads (1×10^9) exposure to eight samples of mordenite (four hydrogen and four silver) was completed. Gas analysis for NO and NO_2 of the air mixture contained in the irradiated sample holders and two control samples, not irradiated, was performed by Dr. D. Davis. His results are given in Table I. The results indicate that irradiation does not seem to affect the NO and NO_2 content of the gas mixture.

Other measurements and analyses of the samples exposed to 10^9 rads are being made; no results are available to report at this time.

II. Crushness Measurements

Crushness measurements on the samples which were irradiated to 1×10^8 rads were made available by Dr. J. Benzel. His results are given in Table II. Comparison of these results with previously reported results indicate that the crushability of the pellets decreased a little, not only for the irradiated samples but also for the unirradiated samples. The density of the same zeolite as reported last month, increased slightly. Dr. Benzel speculates that the increase in hardness, or decrease in crushability, is due to the gas mixture, i.e., the addition of 1000 ppm NO_2 and 1000 ppm O_3. What chemical reaction is responsible for this apparent change is not known at this point.

III. Pressure and Temperature Monitoring

Four sample holders were instrumented to monitor the pressure inside the tubes and temperature in the surface of the tubes. A typical sample of pressure and temperature versus time (date when reading was made) is shown in Figure 1. Obviously not much is happening here.

IV. Certification of Dose and Dose Rates

The appended formal letter of certification for total dose and dose rates for exposures of 1×10^{8} and 1×10^{9} rads is enclosed for your records.

If you have any questions please let me know.

With best wishes.

Sincerely yours,

R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

TABLE I

Gas Analysis for NO and NO_2 from Sample Holders

Irradiated to 1×10^9 Rads

Sample Holder #	Contents	Radiation Dose (Rads)	NO PPB[a]	NO_2 PPB
13	Dry Ag Zeolite + Gas	1.E + 9	19	440
14	Dry Ag Zeolite + Gas	1.E + 9	7	310
15	Sat Ag Zeolite + Gas	1.E + 9	12	180
16	Sat Ag Zeolite + Gas	1.E + 9	6	250
17	Dry H Zeolite + Gas	1.E + 9	11	240
18	Dry H Zeolite + Gas	1.E + 9	6	290
19	Sat H Zeolite + Gas	1.E + 9	26	270
20	Sat H Zeolite + Gas	1.E + 9	30	80
45	Dry H Zeolite + Gas	Not irradiated[b]	31	90
47	Sat H Zeolite + Gas	Not irradiated	46	50

[a]Measurement uncertainty: NO = $\pm$ 6PPB; NO_2 = $\pm$ 40 PPB

[b]Sample holders Nos. 45 and 47 were control samples filled with the same gas at the same time as the others but were not irradiated.

Table II
Mordenite Crushness Results
Irradiated to 1 x 10^8 Rads

	Crushing Load (kgf)				
Mordenite Type	Ag*	Ag	Ag	H	H
Gas Condition	Dry	Dry	Dry	Dry	Dry
Sample Holder #	5	6	41	9	10
Exposure, Rads	10^8	10^8	0	10^8	10^8
	9.70	8.64	10.24	4.24	5.89
	7.28	11.04	12.51	5.17	10.18
	4.52	6.01	12.86	5.58	5.30
	9.77	9.98	7.01	6.07	4.31
	8.36	10.09	6.55	6.26	5.39
	16.06	7.17	10.58		
	3.53	3.23	9.63		
	7.12				
	3.45				
$\bar{x}$	7.754	8.023	9.911	5.464	6.214
S	3.947	2.749	2.443	0.806	2.290
80% Spread	2.702 - 12.806	4.504 - 11.542	6.784 - 13.038	4.432 - 6.496	2.251 - 10.177
95% Reliability	5.175 - 10.333	5.987 - 10.059	8.101 - 11.720	4.758 - 6.170	4.207 - 8.221

*The total number of mordenite pellets varied from sample holder to sample holder because the size of the pellets varied greatly. Consequently the number of pellets in each sample holder varied.

Table II Continued

Mordenite Type	Ag	Ag	Ag	H	H
Gas Condition	Saturated	Saturated	Saturated	Saturated	Saturated
Sample Holder #	43	7	8	11	12
Exposure, Rads	0	10^8	10^8	10^8	10^8
	5.31	6.34	4.33	3.18	4.87
	12.07	4.63	7.20	3.87	7.89
	12.06	8.27	11.24	4.45	11.15
	12.36	7.97	3.63	4.59	9.07
	7.93	3.67	11.14		8.38
		8.05	11.04		
		5.91	8.54		
		6.39			
		2.46			
$\bar{x}$	9.946	5.966	8.160	4.023	8.272
S	3.177	2.041	3.238	0.642	2.272
80% Spread	5.879 - 14.013	3.354 - 8.578	4.015 - 12.305	3.201 - 4.845	5.364 - 11.180
95% Reliability	7.161 - 12.731	4.633 - 7.299	5.761 - 10.559	3.394 - 4.652	6.281 - 10.263

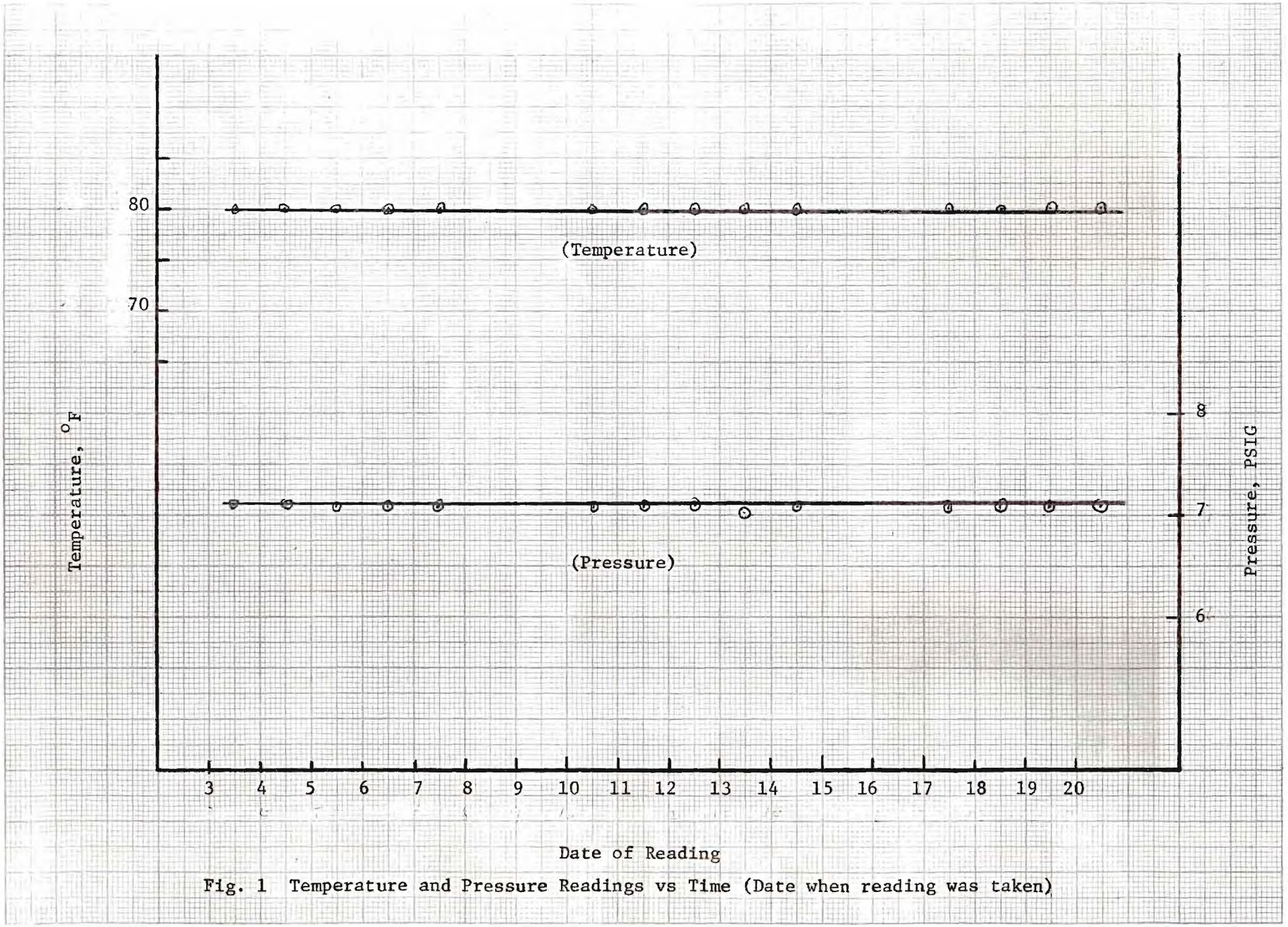


Fig. 1 Temperature and Pressure Readings vs Time (Date when reading was taken)

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

January 18, 1985

Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Attention: Dr. Paul R. Monson Reference: AX-0654697
A-60-603

Gentlemen:

The items covered by the above number have been irradiated in accordance with quality assurance requirements using Cobalt 60 (gamma energies 1.173 Mev, 1.332 Mev) to the total dose requested.

We certify the specifics of the irradiation as follows:

Irradiation Periods:	Intervals between November 13, 1984 and December 21, 1984 as shown on the enclosed Gamma Irradiation Log Sheets
Dose Rates:	See Log Sheets
Total Dose:	See Log Sheets
Dosimetry:	Victoreen Radocon Model 500B-1 Integrating/Rate Electrometer System with ionization chamber probe. Calibration by Victoreen traceable to NBS Cobalt-60.

Calculations, a sketch, and photographs of the arrangement are enclosed. Please let us know if any additional information is needed.

Yours truly,

Dr. R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Enclosures

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

Client: Savannah River NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^8

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
11-13-84	11-15-84				
1135	1400	50.41	1.31×10^6	6.60×10^7	6.60×10^7
11-15-84	11-16-84				
1400	1241	22.67	1.50×10^6	3.40×10^7	1.0×10^8

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

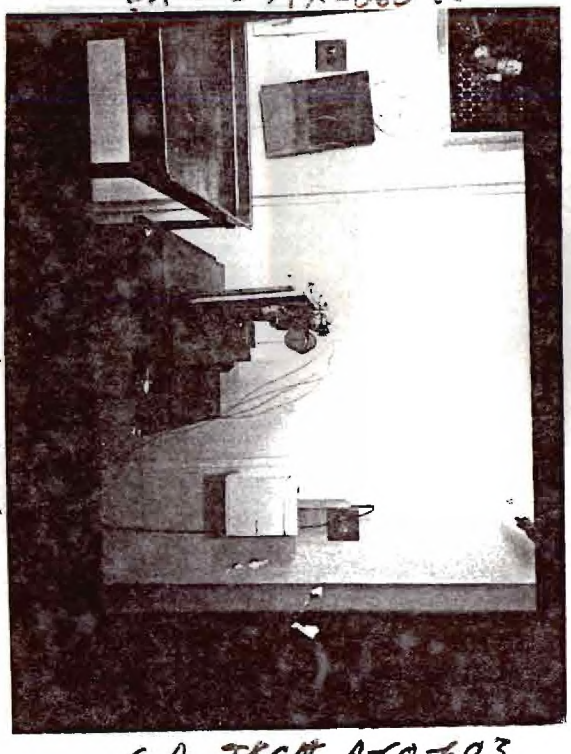
Client: Savannah River NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^9

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
11-16-84	11-21-84				
1500	1430	119.5	1.04×10^6	1.24×10^8	1.24×10^8
11-21-84	12-17/84				
1430	1400	623.5	1.18×10^6	7.35×10^8	8.59×10^8
12-17/84	12-21-84				
1400	1317	95.28	1.48×10^6	1.41×10^8	1.0×10^9

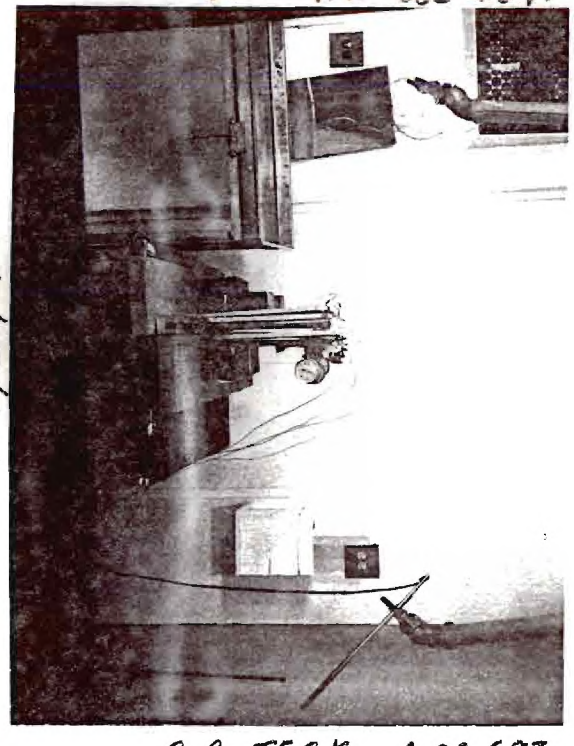
S.R. # AX-065497



8/31/11

G.A. TECH A60603

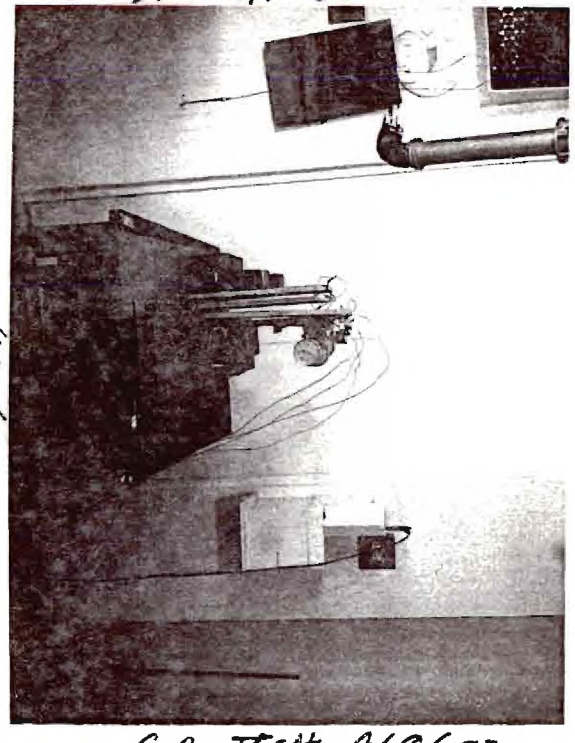
S.R. AX-065497



8/31/11

G.A. TECH A60603

S.R. AX-065497



8/31/11

G.A. TECH A60603

DOSE RATE DETERMINATION

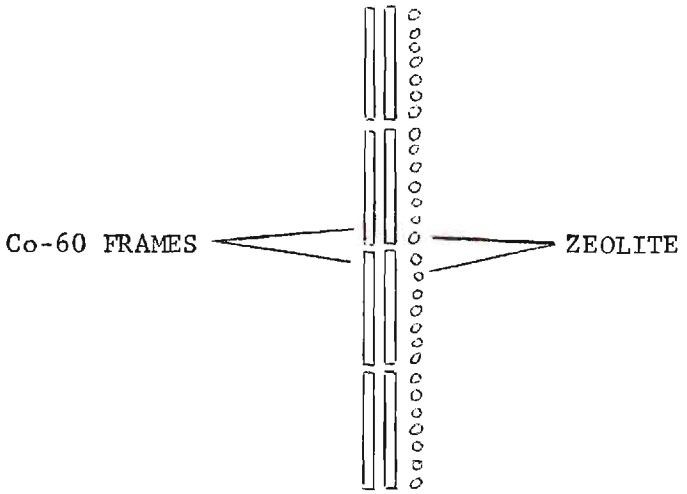
I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to 0 degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
100	0.954	0.869	1.125	60	234.85	1.31×10^6
100	0.954	0.869	1.129	60	268.45	1.50×10^6
100	0.954	0.869	1.129	60	186.35	1.04×10^6
100	0.954	0.869	1.120	60	212.70	1.18×10^6
100	0.954	0.869	1.129	60	264.90	1.48×10^6

Savannah River # AX-0654697

Ga. Tech. # A-60-603



(Not to Scale)

TRACEABILITY DATA

Victoreen Electrometer
Model #500B-1
Serial #340

Calibrated: October 31, 1984
By: Neely Nuclear Research Center
Georgia Institute of Technology
Atlanta, Georgia 30332
Next Calibration Due: October 31, 1985

Instruments Used:

Keithley Picoampere Source
Model #261
Serial #71987

Calibration: March 3, 1984
By: General Electric
5096 Peachtree Road
Chamblee, Georgia 30341
Next Calibration Due: March 3, 1985

General Electric Traceability
N.B.S. #223659 Dated June 23, 1983 Due June 28, 1984
N.B.S. #230753 Dated June 28, 1983 Due June 28, 1984
G.L.I. PET 114/1 Dated August 8, 1983 Due August 8, 1984
Guild Line Instruments
P.O. Box 99
Smith Falls
Ontario, Canada

Hewlett Packard Digital Voltmeter
Model #3460B
Serial #709-00133

Calibration: October 18, 1984
By: Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332
Next Calibration Due: April 18, 1985

Georgia Institute of Technology Traceability
N.B.S. #224381 Dated July 24, 1984 Due January 24, 1985
N.B.S. #223372 Dated July 24, 1984 Due January 24, 1985
N.B.S. #225026 Dated July 24, 1984 Due January 24, 1985
N.B.S. #219868 Dated July 24, 1984 Due January 24, 1985

Victoreen Probe
Model #550-6A
Serial #238

Calibration: April 18, 1984
By: Victoreen, Inc.
10101 Woodland Avenue
Cleveland, Ohio 44104
Next Calibration Due: April 18, 1985

Victoreen Traceability
Test Number DG8118/83
Calibration: September 29, 1983
PTW Chamber Model 30-343
Serial Number N23361-142

A-60-602

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

March 1, 1985

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the months of January and February, 1985 on the project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

I. Density Measurements

Dr. J. Benzel measured the density of the mordenite samples exposed to 1×10^9 rads. He also measured two control hydrogen mordenite samples: one dry and one saturated. The results are given in Table I. If one compares these results with results reported for the months of August and September, 1984, one finds that the density has increased, on the average by 5%. This is true for both silver and hydrogen mordenite. No detectable difference appears between dry and saturated conditions or irradiated and unirradiated samples. Samples numbers 45 and 47 both contained hydrogen mordenite, one dry and one saturated, both were not irradiated, but both had the same apparent density increase as the irradiated samples. The increase in density is small and at this point it not known whether it is real or if it is due to some systematic error. The systematic error will be checked the next time a density measurement is made by measuring the density of some of the original zeolite that is left over and stored in a desicator.

II. Crushing Strength

The crushing loads for the zeolite pellets which were irradiated to 1×10^9 rads are reported in Table II. Comparison of these data with measurements reported in the July and December, 1984 monthly reports shows that the load needed to crush the pellets increased from a value of about 4.9 kgf for the fresh pellets to a range of values of 5.5 to 9.9 kgf for pellets irradiated to 1×10^8 rads and to 5.9 to 11.2 kgf for the pellets irradiated to 1×10^9 rads. Note that the control samples (unirradiated) for both exposures showed tendencies similar to those which were irradiated. The statistical spread in these measurements, however, is large and this point was raised previously. Here again it might be useful to ask Dr. Benzel to measure the crushing strength of the original fresh zeolite.

III. Hardness Measurements

The hardness measurements of samples exposed to 1×10^8 and 1×10^9 rads are given in Table III. Comparison of these data with previous data does not lead to any obvious trend. A closer look and further statistical analysis might be required.

IV. Adsorption and Crystallographic Measurements of Irradiated Samples

Dr. Tudor Thomas performed the crystallographic and adsorption characterizations on the eight samples irradiated to 1×10^9 rads, two control samples and one additional Union Carbide M-8 standard sample. His results are given in Table IV.

Again it appears that:

1. The H-mordenite materials are relatively unaffected by the addition of ozone and NO_2 and exposure to radiation. The retention of crystallinity and oxygen capacity is indicated.

2. Ag-mordenite is much less stable than the H-mordenite to these conditions.

If you have any questions please let me know.

With best wishes.

Sincerely yours,

R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

TABLE I

Density Measurement of Zeolite Samples

Sample #	Type of Mordenite	Exposure, Rads	Gas Condition	Density (gm/cm^3)
13	Ag	10^9	Dry	2.437
14	Ag	10^9	Dry	2.433
15	Ag	10^9	Saturated	2.439
16	Ag	10^9	Saturated	2.440
17	H	10^9	Dry	1.966
18	H	10^9	Dry	1.986
19	H	10^9	Saturated	1.991
20	H	10^9	Saturated	1.980
45	H	0	Dry	1.971
47	H	0	Saturated	1.963

TABLE II

Crushing Load Measurements of Zeolite Samples

	Crushing Load (kgf)				
Type	Ag	Ag	Ag	Ag	H_2
Gas	Dry	Dry	Saturated	Saturated	Dry
Sample Tube	13	14	15	16	17
Rads	10^9	10^9	10^9	10^9	10^9
	10.7	12.1	13.1	13.0	11.0
	14.0	13.3	13.2	9.8	5.9
	8.8	7.3	6.7	6.5	11.6
	8.6	10.3	13.8	9.9	
	14.5	11.1	11.0		
	10.3		9.3		
$\bar{x}$	11.2	10.8	11.2	9.80	9.50
S	2.54	2.27	2.77	2.65	3.13
80% Spread	7.95 - 14.45	7.89 - 13.71	7.65 - 14.75	6.41 - 13.19	5.49 - 13.51
95% Reliability	9.17 - 13.23	8.81 - 12.79	8.98 - 13.42	7.20 - 12.40	5.96 - 13.04

TABLE II (Continued)

	Crushing Load (kgf)				
Type	H_2	H_2	H_2	H_2	H_2
Gas	Dry	Saturated	Saturated	Dry	Saturated
Sample Tube	18	19	20	45	47
Rads	10^9	10^9	10^9	0	0
	14.7	10.3	6.8	8.0	6.4
	7.0	6.6	7.6	3.0	10.2
	10.6	11.3	9.3	4.5	14.0
	8.3		6.0	8.1	9.6
			5.9		
$\bar{x}$	10.15	9.40	7.12	5.90	10.1
S	3.38	2.48	1.40	2.56	3.12
80% Spread	5.82 - 14.48	6.23 - 12.57	5.33 - 8.91	2.62 - 9.18	6.11 - 14.09
95% Reliability	6.84 - 13.46	6.59 - 12.21	5.89 - 8.35	3.39 - 8.41	7.04 - 13.16

TABLE III
Hardness Measurements

Microhardness (Knoop Hardness Number)*

SAMPLE	TYPE	RADS	GAS	KHN	$\bar{X}$	S
5	Ag	10^8	Dry	42.5		
				33.3		
				43.2		
				45.7		
				39.8	40.9	4.74
6	Ag	10^8	Dry	26.2		
				47.2		
				43.9		
				61.9		
				35.3	42.9	13.4
7	Ag	10^8	Saturated	40.6		
				41.6		
				39.0		
				40.8		
				50.8	42.6	4.7
8	Ag	10^8	Saturated	39.0		
				31.0		
				34.6		
				45.5		
				40.9	38.2	5.61
9	H_2	10^8	Dry	76.9		
				90.0		
				124.5		
				73.0		
				104.8	93.8	21.2
10	H_2	10^8	Dry	78.5		
				91.5		
				127.9		
				107.4		
				95.3	100.1	18.6

TABLE III (Continued)

SAMPLE	TYPE	RADS	GAS	KHN	$\bar{X}$	S
11	H_2	10^8	Saturated	95.3		
				96.4		
				53.1		
				79.4		
				74.5	79.7	17.7
12	H_2	10^8	Saturated	48.5		
				84.7		
				66.4		
				81.1		
				62.1	68.6	14.7
13	Ag	10^9	Dry	29.9		
				25.5		
				35.2		
				29.5		
				31.1	30.2	3.48
14	Ag	10^9	Dry	40.3		
				43.2		
				38.3		
				63.6		
				49.6	47.0	10.2
15	Ag	10^9	Saturated	49.6		
				45.7		
				32.2		
				37.8		
				40.8	41.2	6.77
16	Ag	10^9	Saturated	40.5		
				26.5		
				50.4		
				43.0		
				33.9	38.9	9.09

TABLE III (Continued)

SAMPLE	TYPE	RADS	GAS	KHN	$\bar{X}$	S
17	H_2	10^9	Dry	127.9		
				187.9		
				102.3		
				110.8		
				127.0	131.2	33.5
18	H_2	10^9	Dry	215.1		
				91.0		
				130.5		
				87.0		
				65.8	117.9	59.2
19	H_2	10^9	Saturated	126.2		
				108.1		
				89.0		
				92.0		
				116.6	106.4	16.9
20	H_2	10^9	Saturated	125.3		
				110.8		
				92.6		
				105.5		
				130.5	112.9	15.3
41	Ag	0	Dry	38.5		
				40.6		
				46.6		
				44.8		
				49.2	43.9	4.37
43	Ag	0	Saturated	46.4		
				42.9		
				58.0		
				47.0		
				43.0	47.5	6.19
45	H_2	0	Dry	86.5		
				109.5		
				149.1		
				114.4		
				94.8	110.9	24.1

TABLE III (Continued)

SAMPLE	TYPE	RADS	GAS	KHN	$\bar{X}$	S
47	H_2	0	Saturated	215.1		
				108.1		
				160.6		
				97.6		
				136.9	143.7	47.0

*These tests were done on a Tukon Tester manufactured by Wilson Mechanical Instrument Division of American Chain and Cable Company using a 175 gram load. The bottom of the samples were flattened on 180 grit SiC paper so they were stable on the testing stage. The tops of the samples were very lightly ground on 600 grit SiC paper before the hardness measurements were made.

TABLE IV
Adsorption and Crystallographic Properties

Sample Holder #	Contents	Irradiation Dose	% Crystallinity[a,b]	Capacity-wt%[c]
13	dry Ag-M	1×10^9 Rads	17.2	14.0
14	dry Ag-M	1×10^9 Rads	16.5	12.6
15	sat Ag-M	1×10^9 Rads	17.7	13.0
16	sat Ag-M	1×10^9 Rads	15.6	15.3
17	dry H-M	1×10^9 Rads	68.0	23.7
18	dry H-M	1×10^9 Rads	75.6	23.3
19	sat H-M	1×10^9 Rads	77.8	22.7
20	sat H-M	1×10^9 Rads	79.6	23.9
45	dry H-M	0.0	69.8	22.4
47	sat H-M	0.0	74.5	23.6
	Union Carbide H-mordenite	0.0	100.	23.8

[a]X-ray diffraction pattern of the sample relative to a standard H-mordenite sample of 100% crystallinity

[b]All results compare to a commercial H-mordenite powder with 100% crystallinity.

[c]Oxygen adsorption capacity at 75 torr and $-196^{\circ}C$ after activation of $300^{\circ}C$ under vacuum

A-60-602

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Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

April 3, 1985

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the month of March 1985 on the project entitled, "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

The exposure to 5×10^9 rads of 10 samples has recently been completed. Gas analysis is in progress and the rest of the analyses will follow shortly. I don't have any results to report to you yet. I am enclosing some color prints I took of the sample holders. The prints are numbered lightly on the back. Print #1 was taken inside the Hot Cell and the stainless steel cylinders in the center of each irradiation assembly, simulate the actual Co^{60} containing cylinders. The Co^{60} containing cylinders are longer than the "dummys" and produce a uniform field along the entire length of the sample holders which contain the zeolite. Prints #4 and 5 are the only photographs taken from the outside of the Hot Cell through the viewing window with the Co^{60} in place.

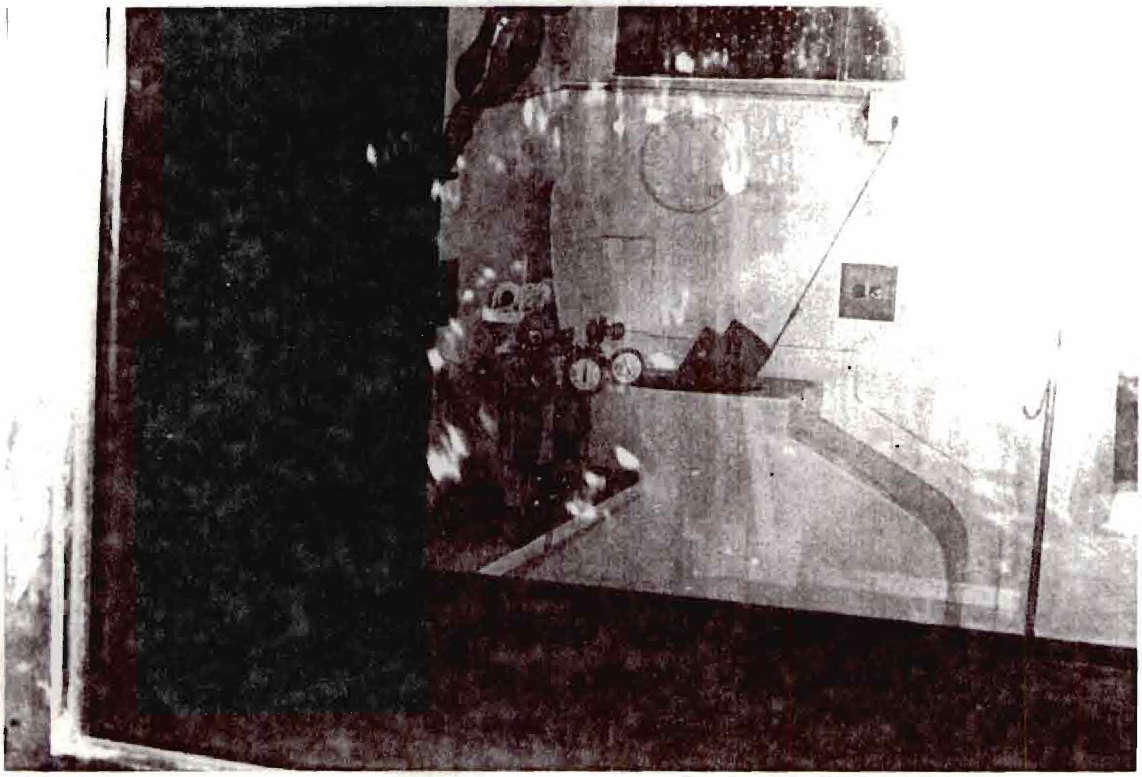
I apologize for the quality of these photographs taken from the outside. This is as good as I can do without filters. If you wish better quality photographs please let me know. For that I need to engage a professional photographer.

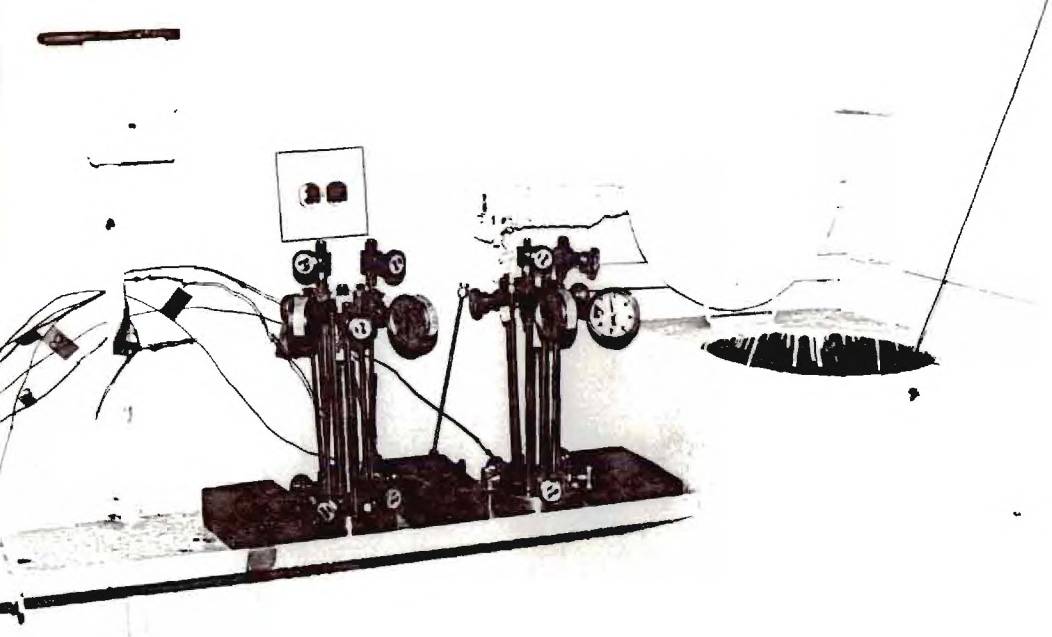
Best personal wishes.

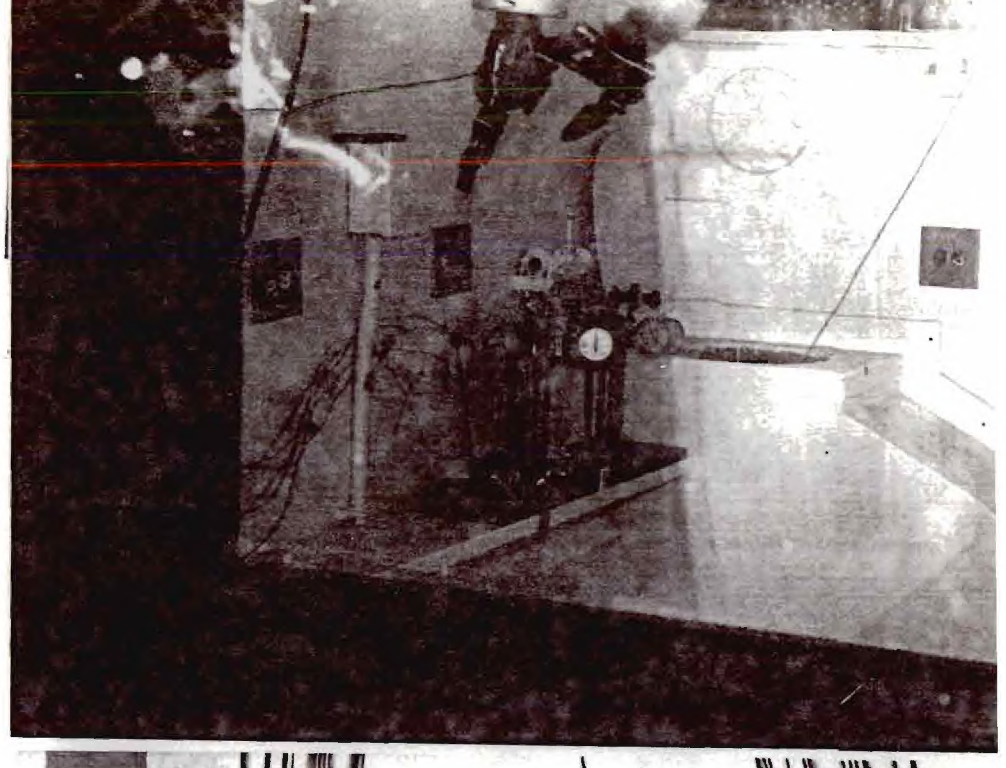
Sincerely yours,

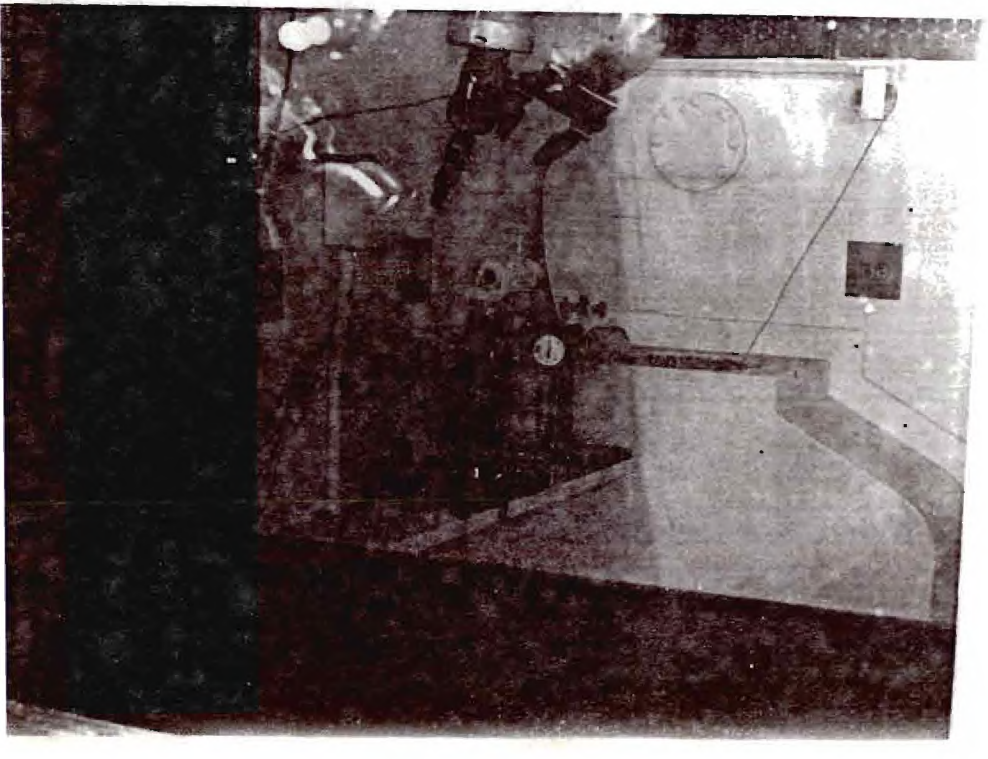
R.A. Karam
Interim Director
Nuclear Research Center

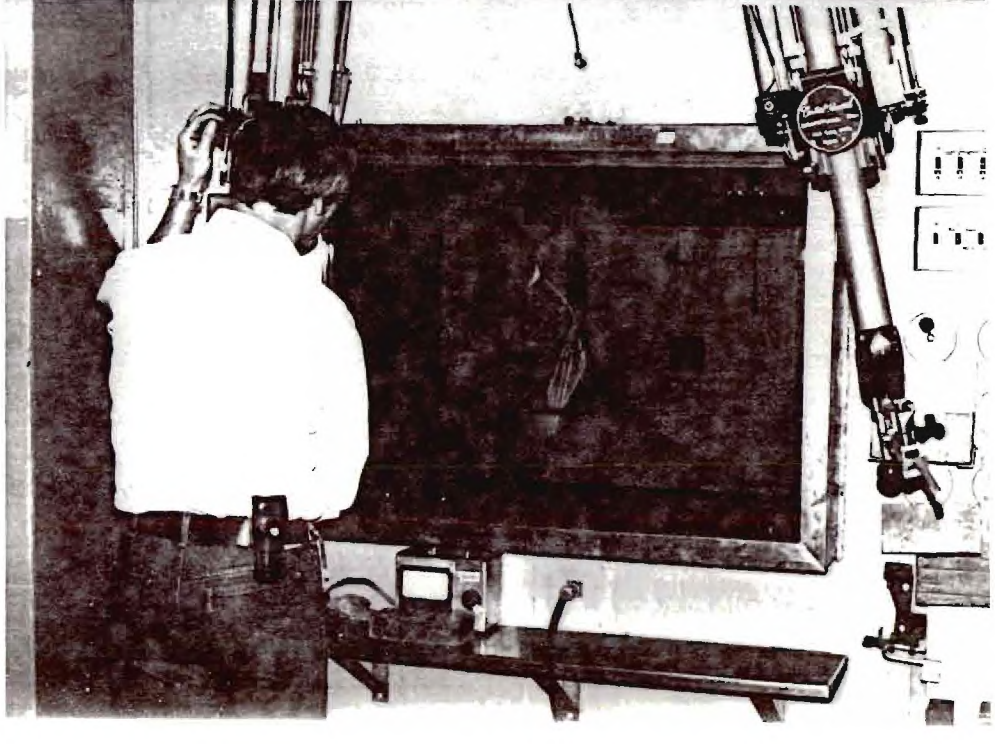
RAK/jlr











A-60-602

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
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GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

May 14, 1985

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the month of April 1985 on the project entitled, "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

I. Gas Analysis

The 5 billion rads (5×10^9) exposure of eight samples of mordenite (four hydrogen and four silver) was completed in early April 1985. Gas analysis for NO and NO_2 of the air mixture contained in the irradiated sample holder and two control samples, not irradiated, was performed by Dr. D. Davis. His results are given in Table I. The results show that increasing the total dose to 5×10^9 rads does not appear to affect the NO and NO_2 content of the gas mixture.

II. Crushing Strength Measurements

Crushing strength measurements of the eight irradiated samples, the two control samples, and two samples (one hydrogen and silver) of the original zeolite not exposed to either NO_2, O_3 or radiation, were measured by Dr. J. Benzel. His results are given in Table II. Comparing these results with those reported in the July 1985 progress report shows that the crushing strength of all zeolites increased, including those for the two samples (A&B) not irradiated and not exposed to NO_2 and O_3. For example the original crushing strength for hydrogen zeolite was 4.943 ± 2.384 kgf compared to 8.34 ± 2.29 kgf now (Sample A). Similarly, the original crushing strength for silver zeolite was 4.726 ± 4.040 kgf compared to 14.3 ± 4.81 kgf now (Sample B). The increase in crushing strength is time dependent but appears to be unrelated to total dose exposure or NO_2 and O_3 treatment. It also appears that the silver zeolite crushing strength is increasing at a faster rate than that of the hydrogen. (Note that the untreated and irradiated mordenite samples (A&B) have been stored in a dessicator all the time.)

III. Adsorption and Crystallographic Measurements

Dr. Tudor Thomas performed the crystallographic and adsorption characterization on the eight samples irradiated to 5×10^9 rads, the two control samples, two samples of original zeolite and one Union Carbide Standard M-8. His results are given in Table III. Comparing these results with previous results tend to show that the adsorption capacity of the silver zeolite is beginning to decrease with increasing the total dose. It would be interesting to see what happens at 1×10^{10} rad level. The hydrogen zeolite on the other hand seems to hold real well.

IV. Certification of Dose and Dose Rates

The appended formal letter of certification for total dose and dose rates for exposure of 5×10^9 rads is enclosed for your records.

If you have any questions please let me know.

Sincerely yours,

R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Enclosure

TABLE I

Gas Analysis for NO and NO_2 from Sample Holders Irradiated to 5×10^9 Rads

Holder #	Contents	Radiation Dose (Rads)	NO PPB[a]	NO_2 PPB
21	Dry Ag Zeolite + Gas	5.E + 9	25	140
22	Dry Ag Zeolite + Gas	5.E + 9	28	150
23	Sat Ag Zeolite + Gas	5.E + 9	18	20
24	Sat Ag Zeolite + Gas	5.E + 9	11	70
25	Dry H Zeolite + Gas	5.E + 9	31	800
26	Dry H Zeolite + Gas	5.E + 9	20	220
27	Sat H Zeolite + Gas	5.E + 9	7	50
28	Sat H Zeolite + Gas	5.E + 9	40	52
46	Dry H Zeolite + Gas	Not irradiated[b]	9	180
48	Sat H Zeolite + Gas	Not irradiated	9	40

[a] Measurement Uncertainty: NO = $\pm$ 2PPB; NO_2 = $\pm$ 20 PPB

[b] Sample holders Nos. 46 and 48 were control samples filled with the same gas at the same time as the others but were not irradiated.

TABLE II

Mordenite Crushness Results
Irradiated to 5×10^9 Rads

	Crushing Load (kgf)					
Mordenite Type	Ag	Ag*	Ag	Ag	H	H
Gas Condition	Dry	Dry	Sat.	Sat.	Dry	Dry
Sample Holder #	21	22	23	24	25	26
Exposure, Rads	5×10^9	5×10^9	5×10^9	5×10^9	5×10^9	5×10^9
	8.8	10.3	8.7	9.3	8.0	10.3
	6.4	11.2	10.4	15.6	8.5	10.8
	11.1	10.7	15.6	8.5	9.4	11.0
	6.3	6.9	8.3	5.8	7.4	8.0
		14.9	10.4			
$\bar{x}$	8.15	10.8	10.7	9.8	8.33	10.0
S	2.28	2.05	2.91	4.15	0.85	1.38
80% Spread	5.23-11.07	7.15-14.45	6.98-14.42	4.49-15.11	7.24-9.42	8.23-11.77
95% Reliability	5.92-10.38	8.30-13.30	8.15-13.25	5.73-13.87	7.50-9.16	8.65-11.35

*The total number of mordenite pellets varied from sample holder to sample holder because the size of the pellets varied greatly. Consequently, the number of pellets in each sample holder varied.

TABLE II CONTINUED

Mordenite Type	H	H	H	H	H	Ag
Gas Condition	Sat	Sat	Dry	Sat	None	None
Sample Holder #	27	28	46	48	A	B
Exposure, Rads	5×10^9	5×10^9	0	0	0	0
	7.6	9.7	10.0	16.6	11.0	18.7
	8.3	10.1	5.6	12.1	9.7	19.0
	7.6	5.0	7.5	15.0	11.5	9.2
	10.5	13.2	5.7	6.0	9.1	12.2
	10.2			7.5	5.2	17.4
					9.0	13.9
					7.9	10.1
					4.9	20.8
					9.0	7.7
					6.1	
$\bar{x}$	8.84	9.5	7.20	11.4	8.34	14.3
S	1.41	3.38	2.06	4.61	2.29	4.81
80% Spread	7.04-10.64	5.17-13.83	4.56-9.84	5.50-17.0	5.41-11.27	8.14-20.46
95% Reliability	7.60-10.08	6.19 12.81	5.18-9.22	7.36-15.44	6.92-9.76	11.16-17.44

TABLE III

Adsorption and Crystallographic Properties

Sample Holder #	Contents	Radiation Dose (Rads)	gms O_2 100 gm Sample	Crystallinity
21	Dry Ag zeolite + Gas	5.0 E + 9	8.1	16
22	Dry Ag zeolite + Gas	5.0 E + 9	11.3	19
23	Sat Ag zeolite + Gas	5.0 E + 9	8.0	20
24	Sat Ag zeolite + Gas	5.0 E + 9	8.3	16
25	Dry H zeolite + Gas	5.0 E + 9	24.1	72
26	Dry H zeolite + Gas	5.0 E + 9	21.3	74
27	Sat H zeolite + Gas	5.0 E + 9	23.3	71
28	Sat H zeolite + Gas	5.0 E + 9	23.8	85
46	Dry H zeolite + Gas	Not irradiated	23.3	84
48	Sat H zeolite + Gas	Not irradiated	26.2	70
A	Special Dry H Sample	Not irradiated	23.9	81
B	Special Dry Ag Sample	Not irradiated	11.3	20
Std	M-8 Union Carbide Standard	Not irradiated	23.2	80

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
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GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

May 9, 1985

Actinide Technology Division
E.I. DuPont de Nemours and Company
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Attention: Dr. Paul R. Monson

Reference: AX-0654697
A-60-603

Gentlemen:

The items covered by the above number have been irradiated in accordance with quality assurance requirements using Cobalt 60 (gamma energies 1.173 Mev, 1.332 Mev) to the total dose requested.

We certify the specifics of the irradiation as follows:

Irradiation Periods:	*Intervals between November 16, 1984 and March 25, 1985 as shown on the enclosed Gamma Irradiation Log Sheets*
Dose Rates:	*See Log Sheet*
Total Dose:	*5.0×10^9 Rads (air)*
Dosimetry:	*Victoreen Model 500B-1 Integrating/Rate Electrometer System with ionization chamber probe. Calibration by Victoreen traceable to NBS Cobalt-60.*

Calculations, a sketch, and photographs of the arrangement are enclosed. Please let us know if any additional information is needed.

Yours truly,

Dr. R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Enclosures

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

Client: *Savannah River Laboratories* NRC Reference: *451024*

Reference: *AX-065-4697* Total Dose: *5×10^9*

Item: *Zeolite Samples* Dose Rate: *Unspecified*

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
11/16/84	*11/21/84*				
1500	*1430*	*119.5*	*1.04×10^6*	*1.24×10^8*	*1.24×10^8*
11/21/84	*12/17/84*				
1430	*1400*	*623.5*	*1.18×10^6*	*7.35×10^8*	*8.59×10^8*
12/17/84	*12/21/84*				
1400	*1317*	*95.28*	*1.48×10^6*	*1.41×10^8*	*1.0×10^9*
12/21/84	*1/8/85*				
1545	*0825*	*424.66*	*1.25×10^6*	*5.30×10^8*	*1.53×10^9*
1/24/85	*1/25/85*				
1607	*0812*	*16.08*	*2.29×10^6*	*3.68×10^7*	*1.56×10^9*
1/31/85	*2/4/85*				
1535	*1309*	*93.56*	*3.74×10^6*	*3.49×10^8*	*1.90×10^9*
2/5/85	*2/7/85*				
1605	*0930*	*41.41*	*3.74×10^6*	*1.54×10^8*	*2.05×10^9*

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

Client: Savannah River NRC Reference: 451024

Reference: AX-065-4697 Total Dose: 5.0×10^9

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
2/7/85	2/8/85				
1407	1327	23.33	3.74×10^6	8.72×10^7	2.13×10^9
2/9/85	2/11/85				
1619	0915	40.93	3.74×10^6	1.53×10^8	2.28×10^9
2/11/85	2/20/85				
1221	1321	217.0	3.74×10^6	8.11×10^8	3.09×10^9
2/27/85	2/28/85				
1625	0925	17.0	3.74×10^6	6.35×10^7	3.15×10^9
2/28/85	3/5/85				
1545	1345	118.0	3.74×10^6	4.41×10^8	3.59×10^9
3/5/85	3/6/85				
1416	1016	20.0	3.74×10^6	7.48×10^7	3.66×10^9
3/6/85	3/12/85				
1630	1115	138.75	3.74×10^6	5.18×10^8	4.17×10^9
3/13/85	3/18/85				
1130	1030	119.0	3.74×10^6	4.45×10^8	4.61×10^9

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

Client: Savannah River NRC Reference: 451024

Reference: AX-0654697 Total Dose: 5.0×10^9

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
3/19/85	3/20/85				
1615	1315	21.0	3.74×10^6	7.85×10^7	4.68×10^9
3/20/85	3/21/85				
1515	1015	19.0	3.74×10^6	7.10×10^7	4.75×10^9
3/22/85	3/25/85				
1304	0755	66.85	3.74×10^6	2.50×10^8	5.0×10^9

-60-602

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

July 24, 1985

Dr. Paul R. Monson
Actinide Technology Division
E.I. DuPont de Nemours and Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Dear Paul:

This is a progress report for the months of May and June, 1985 on the project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses."

I. Gas Analysis

The 10 billion rads (1×10^{10}) exposure of 12 samples of mordenite (six hydrogen and six silver) and two gas only samples were completed in early June 1985. Gas analysis for NO and NO_2 of the air mixture contained in the irradiated samples and four unirradiated samples, was performed by Dr. D. Davis. His results are given in Table I.

II. Density Measurements

Dr. J. Benzel measured the density of samples exposed to doses of 5×10^9 rads and 1×10^{10} rads. The results are given in Tables II and III respectively. The last two samples listed in Table II and also in Table III are original zeolite supplied by Savannah River Laboratory that was stored in a dessicator and not exposed to radiation, NO_2 or ozone. The values listed for these samples provide a reference against which reproducibility can be evaluated. Further analysis of these data will be deferred until the final report.

III. Crushing Strength Measurements

Dr. J. Benzel also measured the crushing strength of the samples exposed to 1×10^{10} rads. The data are given in Table IV.

IV. Hardness Measurements

The microhardness measurements for samples irradiated to 5×10^{9} rads and 1×10^{10} rads are given in Table V.

V. Adsorption and Crystallographic Measurements

Dr. Tudor Thomas measured the adsorption and crystallographic properties of the samples irradiated to 1×10^{10} rads. The results are given in Table VI. Note the standard sample M-8 reported in Table VI is a powder sample whereas the M-8 standards reported in the April progress report was a pellet. That is why the percent crystallinity for the two standards differ.

VI. Certification of Dose and Dose Rates

The approved formal letter of certification for total dose and dose rates for exposure of 1×10^{10} rads is enclosed for your records.

VII. Analysis

No analysis of the data has been attempted up to this point. I recommend that you and other interested personnel from SRL plan to meet with us for perhaps one day here at Georgia Tech to discuss informally what we have learned thus far before I attempt to write the final report. There is one constraint with regard to the meeting: Tudor Thomas will be on vacation August 9-September 9. If you can make it before August 9, 1985 please let me know. With best wishes.

Sincerely,

R.A. Karam

RAK:jlr

Enclosures

TABLE I

Gas Analysis for NO and NO_2 from
Sample Holders Irradiated to $1x10^{10}$ Rads

HOLDER NUMBER	CONTENTS	RADIATION DOSE (Rads)	NO PPB[a]	NO_2 PPB
1	Dry Ag Zeolite + Gas (Gauged)	1.0 E + 10	3	80
2	Sat Ag Zeolite + Gas (Gauged)	1.0 E + 10	21	280
3	Dry H Zeolite + Gas (Gauged)	1.0 E + 10	6400	950
4	Sat H Zeolite + Gas (Gauged)	1.0 E + 10	8	160
29	Dry Ag Zeolite + Gas	1.0 E + 10	7	120
30	Dry Ag Zeolite + Gas	1.0 E + 10	120	520
31	Sat Ag Zeolite + Gas	1.0 E + 10	52	430
32	Sat Ag Zeolite + Gas	1.0 E + 10	70	480
33	Dry H Zeolite + Gas	1.0 E + 10	440	970
34	Dry H Zeolite + Gas	1.0 E + 10	2220	1330
35	Sat H Zeolite + Gas	1.0 E + 10	14300	6000
36	Sat H Zeolite + Gas	1.0 E + 10	5100	3500
37	Dry Gas Only[b]	1.0 E + 10	106000	87000
38	Sat Gas Only[b]	1.0 E + 10	72000	63000
39	Dry Gas Only[b]	Not Irradiated	10100	24000
40	Sat Gas Only[b]	Not Irradiated	3500	9100
42	Dry Ag Zeolite + Gas[b]	Not Irradiated	501	2500
44	Sat Ag Zeolite + Gas[b]	Not Irradiated	280	1700

[a] Measurement uncertainty, according to D. Davis, is $\pm$25% due to high NO content.

[b] Control samples

TABLE II

Density Measurement of Zeolite Samples

Sample	Type	Rads	Gas	Density (gm/cm^2)
21	Ag	$5x10^9$	Dry	2.343
22	Ag	$5x10^9$	Dry	2.356
23	Ag	$5x10^9$	Saturated	2.374
24	Ag	$5x10^9$	Saturated	2.305
25	H_2	$5x10^9$	Dry	1.877
26	H_2	$5x10^9$	Dry	1.903
27	H_2	$5x10^9$	Saturated	1.914
28	H_2	$5x10^9$	Saturated	1.897
46	H_2	0	Dry	1.942
48	H_2	0	Saturated	1.918
Fresh	H_2	0	None	1.875
Fresh	Ag	0	None	2.350

TABLE III

Density Measurement of Zeolite Samples

Sample	Type	Rads	Gas	Density (gm/cm^2)
1	Ag	1×10^{10}	Dry	2.403
2	Ag	1×10^{10}	Saturated	2.386
3	H_2	1×10^{10}	Dry	1.908
4	H_2	1×10^{10}	Saturated	1.914
29	Ag	1×10^{10}	Dry	2.398
30	Ag	1×10^{10}	Dry	2.452
31	Ag	1×10^{10}	Saturated	2.365
32	Ag	1×10^{10}	Saturated	2.373
33	H_2	1×10^{10}	Dry	1.907
34	H_2	1×10^{10}	Dry	1.921
35	H_2	1×10^{10}	Saturated	1.962
36	H_2	1×10^{10}	Saturated	1.919
42	Ag	0	Dry	2.391
44	Ag	0	Saturated	2.404
Fresh	Ag	0	None	2.283
Fresh	H_2	0	None	1.942

TABLE IV

Crushness Results of Mordenite Samples Irradiated to $1x10^{10}$ Rads

	Crushing Load (kgf)						
	Ag Dry	Ag Saturated	H_2 Dry	H_2 Saturated	Ag Dry	Ag Dry	Ag Saturated
le Tube	1	2	3	4	29	30	31
	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$
	16.7	13.9	5.8	9.7	18.0	10.88	14.7
	20.6	8.8	12.1	8.0	9.1		20.1
	7.9	11.3	7.2	8.8	10.2		8.9
		16.2	15.3	6.6	13.3		
	15.1	12.6	10.1	8.28	9.0	*	14.6
	6.51	3.20	4.39	1.31	3.82	*	5.60
Spread	6.74-	8.45-	4.48-	6.60-	7.03-		7.40-
	23.4	16.6	15.7	9.96	16.8	*	21.7
Reliability	7.73-	9.46-	5.80-	7.00-	8.55-		8.26-
	22.5	15.7	14.4	9.56	15.2	*	20.9

y one sample available for testing.

TABLE IV (CONTINUED)

	Crushing Load (kgf)					
Type	Ag	H_2	H_2	H_2	H_2	Ag
Gas	Saturated	Dry	Dry	Saturated	Saturated	Dry
Sample Tube	32	33	34	35	36	42
Rads	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	$1x10^{10}$	0
	12.2	9.1	6.8	11.1	8.0	6.0
	14.8	8.0	7.4	8.6	10.1	11.0
	9.4	10.1	12.5	7.7	9.5	7.5
	14.1	8.4	8.6		12.6	
		12.7	7.9			
$\bar{x}$	12.6	9.66	8.64	9.10	10.1	8.17
S	2.41	1.88	2.26	1.71	1.92	2.57
80% Spread	9.55-15.7	7.25-12.1	5.75-11.5	6.91-11.3	7.59-12.5	4.88-11.9
95% Reliability	10.2-15.0	8.01-11.3	6.66-10.6	7.16-11.0	8.22-12.0	5.26-11.1

TABLE IV (CONTINUED)

	Crushing Load (kgf)				
Type	Ag	H_2	H_2 (1)	Ag	Ag (1)
Gas	Saturated	None	None	None	None
Sample Tube	44	Fresh	Fresh	Fresh	Fresh
Rads	0	0	0	0	0
	6.6	6.6	3.3	10.1	7.0
	7.1	7.8	4.6	8.3	5.1
	17.6	6.5	2.7	12.2	13.0
		7.3	3.6	17.3	13.2
		13.8	5.7	13.9	12.4
		13.0	5.8	9.3	9.6
		5.3	3.1	12.0	9.5
		9.1	7.4	11.0	3.9
		8.5	6.9	8.9	8.6
		8.6	3.4	11.6	5.8
$\bar{x}$	10.4	8.65	4.65	11.5	8.81
S	6.21	2.75	1.69	2.67	3.35
80% Spread	2.48-18.4	5.13-12.2	2.48-6.81	8.04-14.9	4.52-13.1
95% Reliability	3.37-17.4	6.95-10.4	3.60-5.70	9.85-13.2	6.73-10.9

(1) Cross-head speed changed form 0.05 cm/min to 0.005 cm/min.

TABLE V

Microhardness Results of Mordenite Samples Irradiated to 5×10^9 and 1×10^{10} Rads

Sample	Type	Rads	Gas	KHN	$\bar{X}$	S
1	Ag	10^{10}	Dry	43.3		
				32.1		
				44.2		
				45.6		
				43.3	41.7	5.45
2	Ag	10^{10}	Saturated	38.3		
				63.9		
				47.9		
				46.2		
				33.2	45.9	11.7
3	H_2	10^{10}	Dry	105.1		
				119.3		
				66.1		
				69.0		
				79.2	87.7	23.4
4	H_2	10^{10}	Saturated	126.8		
				110.8		
				114.4		
				129.3		
				128.7	122.0	8.72
21	Ag	5×10^9	Dry	35.5		
				36.5		
				33.0		
				36.8		
				47.3	37.9	5.59
22	Ag	5×10^9	Dry	44.2		
				36.2		
				39.8		
				51.5		
				45.3	43.4	5.80

TABLE V (CONTINUED)

Sample	Type	Rads	Gas	KHN	$\bar{X}$	S
23	Ag	$5x10^9$	Saturated	34.1		
				24.9		
				34.2		
				38.0		
				31.3	32.5	4.87
24	Ag	$5x10^9$	Saturated	39.7		
				48.3		
				66.3		
				30.6		
				51.5	47.3	13.4
25	H_2	$5x10^9$	Dry	106.7		
				124.7		
				105.1		
				106.9		
				133.9	115.5	13.1
26	H_2	$5x10^9$	Dry	101.1		
				69.1		
				72.7		
				107.6		
				103.0	90.7	18.3
27	H_2	$5x10^9$	Saturated	47.1		
				60.2		
				96.7		
				44.2		
				77.0	65.0	21.9
28	H_2	$5x10^9$	Saturated	94.6		
				95.7		
				63.1		
				89.9		
				86.2	85.9	13.3

TABLE V (CONTINUED)

Sample	Type	Rads	Gas	KHN	$\bar{X}$	S
29	Ag	10^{10}	Dry	38.0		
				37.5		
				36.4		
				38.2		
				49.9	40.0	5.58
30	Ag	10^{10}	Dry	No Sample Available for testing.		
31	Ag	10^{10}	Saturated	47.5		
				39.2		
				43.7		
				54.0		
				49.1	46.7	5.59
32	Ag	10^{10}	Saturated	40.6		
				44.9		
				63.2		
				28.3		
				58.5	47.1	14.1
33	H_2	10^{10}	Dry	76.5		
				89.5		
				129.9		
				109.4		
				93.3	99.7	20.6
34	H_2	10^{10}	Dry	96.1		
				102.2		
				63.9		
				85.2		
				80.3	85.5	14.9
35	H_2	10^{10}	Saturated	131.3		
				177.8		
				105.7		
				114.2		
				130.4	131.9	27.8

TABLE V (CONTINUED)

Sample	Type	Rads	Gas	KHN	$\bar{X}$	S
36	H_2	10^{10}	Saturated	81.9		
				109.8		
				95.0		
				78.1		
				124.9	97.9	19.5
42	Ag	0	Dry	46.7		
				106.7		
				21.1		
				29.6		
				45.8	50.0	33.5
44	Ag	0	Saturated	43.8		
				40.4		
				25.9		
				31.5		
				35.6	35.4	7.1
46	H_2	0	Dry	160.8		
				90.3		
				129.8		
				87.7		
				96.5	113.0	31.6
48	H_2	0	Saturated	91.8		
				87.3		
				110.4		
				99.4		
				90.4	95.9	9.26
Fresh	Ag	0	None	41.2		
				26.5		
				51.1		
				37.5		
				28.4	36.9	10.0

TABLE V (CONTINUED)

Sample	Type	Rads	Gas	KHN	$\bar{X}$	S
Fresh	H_2	0	None	142.3		
				97.3		
				136.6		
				107.8		
				196.8	136.2	38.8

*These tests were done on a Tukon Tester manufactured by Wilson Mechanical Instrument Division of American Chain and Cable Company using a 175 gram load. The bottom of the samples were flattened on 180 grit SiC paper so they were stable on the testing stage. The tops of the samples were very lightly ground on 600 grit SiC paper before the hardness measurements were made.

TABLE VI

Adsorption and Crystallographic Measurements of Samples Irradiated to $1x10^{10}$ Rads

SAMPLE HOLDER	CONTENTS	RADIATION DOSE (RADS)	gms O_2 100 gm SAMPLE	% CRYSTALLINITY
1	Dry Ag zeolite + Gas (Gauged)	1.0 E + 10	16.1	20.9
2	Sat Ag zeolite + Gas (Gauged)	1.0 E + 10	16.7	22.2
3	Dry H zeolite + Gas (Gauged	1.0 E + 10	23.7	78.3
4	Sat H zeolite + Gas (Gauged)	1.0 E + 10	23.8	83.1
29	Dry Ag zeolite + Gas	1.0 E + 10	11.2	18.1
30	Dry Ag zeolite + Gas	1.0 E + 10	14.0	18.8
31	Sat Ag zeolite + Gas	1.0 E + 10	7.8	19.7
32	Sat Ag zeolite + Gas	1.0 E + 10	14.0	15.9
33	Dry H zeolite + Gas	1.0 E + 10	23.9	80.0
34	Dry H zeolite + Gas	1.0 E + 10	27.9	79.6
35	Sat H zeolite + Gas	1.0 E + 10	23.3	86.5
36	Sat H zeolite + Gas	1.0 E + 10	23.6	84.2
42	Dry Ag zeolite + Gas	Not Irradiated	12.0	20.6
44	Dry Ag zeolite + Gas	Not Irradiated	12.7	21.3
STD	M-8 powder	Not Irradiated	28.0	100.0

Georgia Institute of Technology
Neely Nuclear Research Center
Atlanta, Georgia 30332
(404) 894-3600

GEORGIA TECH 1885-1985

DESIGNING TOMORROW TODAY

June 26, 1985

Actinide Technology Division
E.I. DuPont de Nemours & Company, Inc.
Atomic Energy Division
Savannah River Laboratory
Aiken, South Carolina 29808

Attention: Dr. Paul R. Monson Reference: AX-0654697
A-60-603

Gentlemen:

The items covered by the above number have been irradiated in accordance with quality assurance requirements using Cobalt 60 (gamma energies 1.173 Mev, 1.332 Mev) to the total dose requested.

We certify the specifics of the irradiation as follows:

Irradiation Periods:	Intervals between November 16, 1984 and June 11, 1985 as shown on the enclosed Gamma Irradiation Log Sheets
Dose Rates:	See Log Sheets
Total Dose:	1.0×10^{10} Rads (air)
Dosimetry:	Victoreen Model 500B-1 Integrating/Rate Electrometer System with ionization chamber probe. Calibration by Victoreen traceable to NBS Cobalt-60.

Calculations, a sketch, and photographs of the arrangement are enclosed. Please let us know if any additional information is needed.

Yours truly,

Dr. R.A. Karam
Interim Director
Nuclear Research Center

RAK/jlr

Enclosures

GEORGIA INSTITUTE OF TECHNOLOGY

Nuclear Research Center
900 Atlantic Drive, N.W.
Atlanta, Georgia 30332
(404) 894-3608

GAMMA IRRADIATION LOG

Client: Savannah River Laboratory NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^{10}

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
11/16/84	11/21/84				
1500	1430	119.5	1.04×10^{6}	1.24×10^{8}	1.24×10^{8}
11/21/84	12/17/84				
1430	1400	623.5	1.18×10^{6}	7.35×10^{8}	8.59×10^{8}
12/17/84	12/21/84				
1400	1317	95.28	1.48×10^{6}	1.41×10^{8}	1.0×10^{9}
12/21/84	1/8/85				
1545	0825	424.66	1.25×10^{6}	5.30×10^{8}	1.53×10^{9}
1/31/85	2/4/85				
1536	1310	93.56	2.38×10^{6}	2.22×10^{8}	1.75×10^{9}
2/5/85	2/7/85				
1606	0931	41.41	2.38×10^{6}	9.85×10^{7}	1.84×10^{9}
2/7/85	2/8/85				
1408	1328	23.33	2.38×10^{6}	5.55×10^{7}	1.89×10^{9}
2/9/85	2/11/85				
1620	0916	40.93	2.38×10^{6}	9.74×10^{7}	1.98×10^{9}

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Atlanta, Georgia 30332
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GAMMA IRRADIATION LOG

Client: Savannah River Laboratory NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^{10}

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
2/11/85	2/20/85				
1222	1322	217.0	2.38×10^{6}	5.16×10^{8}	2.49×10^{9}
2/27/85	2/28/85				
1626	0926	17.0	2.38×10^{6}	4.04×10^{7}	2.53×10^{9}
2/28/85	3/5/85				
1546	1346	118.0	2.38×10^{6}	2.80×10^{8}	2.81×10^{9}
3/5/85	3/6/85				
1417	1017	20.0	2.38×10^{6}	4.76×10^{7}	2.85×10^{9}
3/6/85	3/12/85				
1631	1116	138.75	2.38×10^{6}	3.30×10^{8}	3.18×10^{9}
3/13/85	3/18/85				
1131	1031	119.0	2.38×10^{6}	2.83×10^{8}	3.46×10^{9}

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GAMMA IRRADIATION LOG

Client: Savannah River Laboratory

NRC Reference: 451024

Reference: AX-0654697

Total Dose: 1.0×10^{10}

Item: Zeolite Samples

Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
3/19/85	3/25/85				
1616	0755	135.65	2.38×10^{6}	3.22×10^{8}	3.78×10^{9}
3/25/85	3/27/85				
1620	1020	42.0	3.91×10^{6}	1.64×10^{8}	3.94×10^{9}
3/27/85	4/1/85				
1525	1525	120.0	3.91×10^{6}	4.69×10^{8}	4.40×10^{9}
4/3/85	4/3/85				
1030	1230	2.0	3.91×10^{6}	7.82×10^{6}	4.40×10^{9}
4/5/85	4/10/85				
1555	1125	115.5	3.91×10^{6}	4.51×10^{8}	4.85×10^{9}
4/10/85	4/11/85				
1612	0940	17.46	3.91×10^{6}	6.82×10^{7}	4.91×10^{9}
4/11/85	4/15/85				
1325	1525	98.0	3.91×10^{6}	3.83×10^{8}	5.29×10^{9}
4/15/85	4/16/85				
1610	1010	18.0	3.91×10^{6}	7.03×10^{7}	5.36×10^{9}

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Atlanta, Georgia 30332
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GAMMA IRRADIATION LOG

Client: Savannah River Laboratory NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^{10}

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
4/16/85	4/17/85				
1525	1345	22.33	3.91×10^{6}	8.73×10^{7}	5.44×10^{9}
4/17/85	4/23/85				
1620	1105	138.75	3.91×10^{6}	5.42×10^{8}	5.98×10^{9}
4/23/85	4/26/85				
1610	1200	67.83	3.91×10^{6}	2.65×10^{8}	6.24×10^{9}
4/26/85	4/29/85				
1616	1153	66.61	3.91×10^{6}	2.60×10^{8}	6.50×10^{9}
NOTE:	Time Change Occurred During Last Period				
4/30/85	5/6/85				
1555	1100	139.08	3.91×10^{6}	5.43×10^{8}	7.04×10^{9}
5/7/85	5/8/85				
0900	0830	23.5	3.91×10^{6}	9.18×10^{7}	7.13×10^{9}
5/8/85	5/9/85				
1040	0920	22.66	3.91×10^{6}	8.86×10^{7}	7.21×10^{9}

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GAMMA IRRADIATION LOG

Client: Savannah River Laboratory NRC Reference: 451024

Reference: AX-0654697 Total Dose: 1.0×10^{10}

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
5/9/85	5/15/85				
1125	0735	140.16	3.90×10^{6}	5.46×10^{8}	7.75×10^{9}
5/15/85	5/16/85				
1100	1130	24.50	3.90×10^{6}	9.55×10^{7}	7.84×10^{9}
5/16/85	5/18/85				
1445	0845	42.00	3.90×10^{6}	1.63×10^{8}	8.00×10^{9}
5/18/85	5/20/85				
0925	0825	47.00	3.90×10^{6}	1.83×10^{8}	8.18×10^{9}
5/21/85	5/22/85				
1535	0915	17.66	3.90×10^{6}	6.88×10^{7}	8.24×10^{9}
5/22/85	5/23/85				
1115	0922	22.11	3.90×10^{6}	8.62×10^{7}	8.32×10^{9}
5/23/85	5/28/85				
1510	0920	114.16	3.90×10^{6}	4.45×10^{8}	8.76×10^{9}
5/28/85	5/31/85				
1120	1350	74.5	3.90×10^{6}	2.90×10^{8}	9.05×10^{9}

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GAMMA IRRADIATION LOG

Client: Savannah River Laboratory NRC Reference: 451024

Reference: AX-0654-697 Total Dose: 1.0×10^{10}

Item: Zeolite Samples Dose Rate: Unspecified

Start	Stop	Lapsed Hours	Dose Rate Rads/hour	Total Dose Rads	Cumulative Dose - Rads
5/31/85	6/5/85				
1545	0957	114.2	3.90×10^{6}	4.45×10^{8}	9.49×10^{9}
6/5/85	6/7/85				
1110	0810	45.0	3.90×10^{6}	1.75×10^{8}	9.66×10^{9}
6/7/85	6/7/85				
0835	1541	7.1	3.90×10^{6}	2.76×10^{7}	9.68×10^{9}
6/7/85	6/10/85				
1553	0753	64.0	3.90×10^{6}	2.49×10^{8}	9.92×10^{9}
6/10/85	6/10/85				
0930	1320	3.83	3.90×10^{6}	1.49×10^{7}	9.93×10^{9}
6/10/85	6/11/85				
1415	0815	18.0	3.90×10^{6}	7.0×10^{7}	1.0×10^{10}

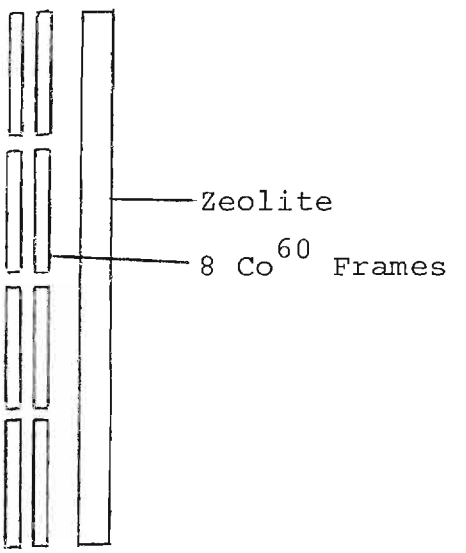
DOSE RATE DETERMINATION

I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to 0 degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
10	0.954	0.869	1.129	60	183.35	1.04×10^6

SRL #: AX-0654697
GT #: A-60-603



(Not to Scale)

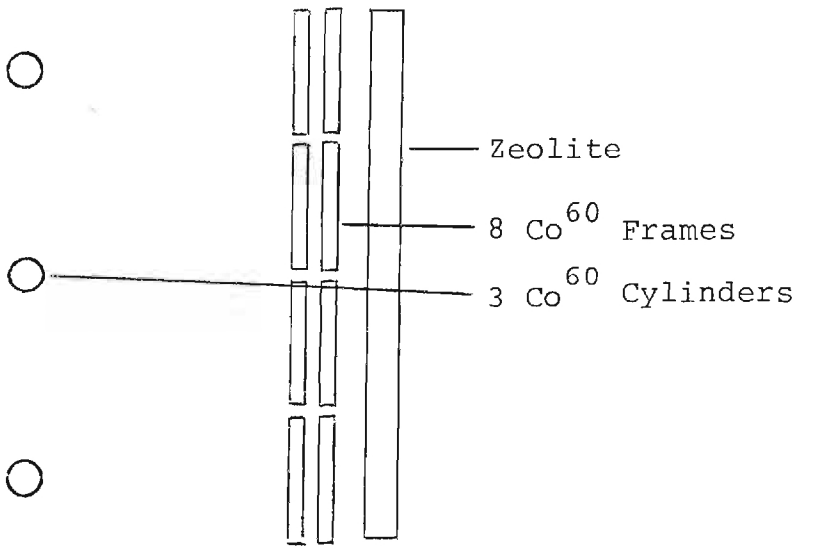
DOSE RATE DETERMINATION

I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to Ø degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
ØØ	Ø.954	Ø.869	1.12Ø	6Ø	212.7Ø	1.18×10^{6}

SRL #: AX-0654697
GT #: A-60-603



(Not to Scale)

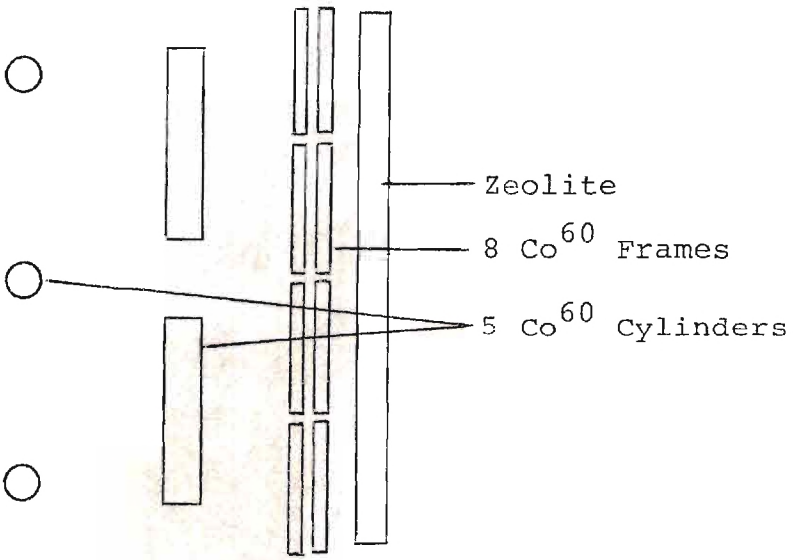
DOSE RATE DETERMINATION

I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to Ø degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
Ø0	Ø.954	Ø.869	1.129	60	264.9Ø	1.48×10^{6}

SRL #: AX-0654697
GT #: A-60-603



(Not to Scale)

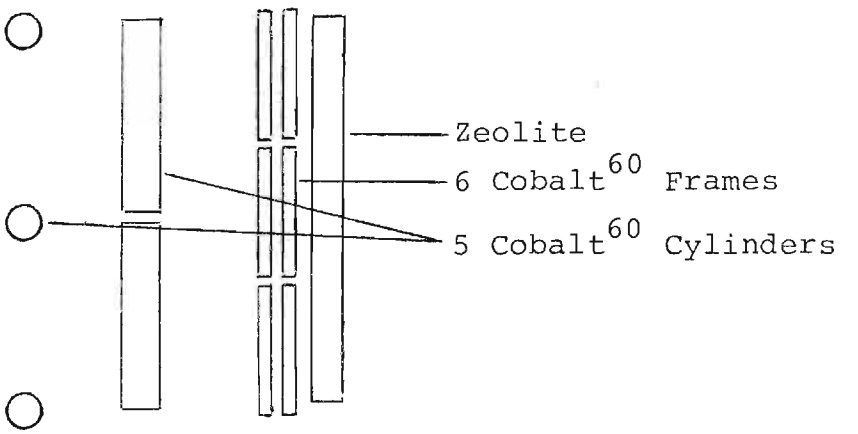
DOSE RATE DETERMINATION

I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to 0 degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
00	0.954	0.869	1.135	60	221.66	1.25×10^{6}

SRL #: AX-0654697
GT #: A-60-603



(Not to Scale)

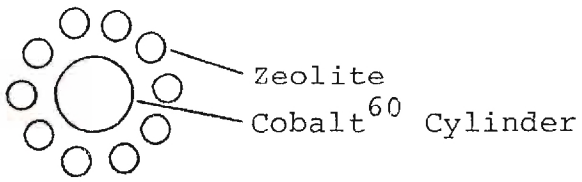
DOSE RATE DETERMINATION

I x P x D x A x T x R = Dose rate, rads/hr (air equivalent)

I = Electrometer high level conversion
P = Probe efficiency
D = Dose conversion, roentgen to rad
A = Temperature & Pressure correction to standardize to 0 degree Celsius and 760 mm Hg
T = Time conversion, min. to hr.
R = Electrometer reading

I	P	D	A	T	R	Dose rate, rads/hr
00	1.036	0.869	1.145	60	385.70	2.38×10^{6}
00	1.036	0.869	1.122	60	646.65	3.91×10^{6}
00	1.036	0.869	1.130	60	639.98	3.90×10^{6}

SRL #: AX-0654697
GT #: A-60-603



(Not to Scale)

TRACEABILITY DATA

Victoreen Electrometer
Model #500B-1
Serial #340

Calibrated: October 31, 1984
By: Neely Nuclear Research Center
Georgia Institute of Technology
Atlanta, Georgia 30332
Next Calibration Due: October 31, 1985

Instruments Used:
Keithley Picoampere Source
Model #261
Serial #71987

Calibration: March 3, 1984
By: General Electric
5096 Peachtree Road
Chamblee, Georgia 30341
Next Calibration Due: March 3, 1985

General Electric Traceability
N.B.S. #223559 Dated June 23, 1983 Due June 28, 1984
N.B.S. #230753 Dated June 28, 1983 Due June 28, 1984
G.L.I. PET 114/1 Dated August 8, 1983 Due August 8, 1984
Guild Line Instruments
P.O. Box 99
Smith Falls
Ontario, Canada

Hewlett Packard Digital Voltmeter
Model #3460B
Serial #709-00133

Calibration: October 18, 1984
By: Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332
Next Calibration Due: April 18, 1985

Georgia Institute of Technology Traceability
N.B.S. #224381 Dated July 24, 1984 Due January 24, 1985
N.B.S. #223372 Dated July 24, 1984 Due January 24, 1985
N.B.S. #225026 Dated July 24, 1984 Due January 24, 1985
N.B.S. #219868 Dated July 24, 1984 Due January 24, 1985

Victoreen Probe
Model #550-6A
Serial #25[illegible]

Calibration: November 14, 1984
By: Victoreen, Inc.
19101 Woodland Avenue
Cleveland, Ohio 44104
Next Calibration Due: November 14, 1985

Victoreen Traceability
Test Number DG8118/83
Calibration: September 29, 1983
PTW Chamber Model 30-343
Serial Number N23361-142

Victoreen Probe
Model #550-6A
Serial #238

Calibration: April 18, 1984
By: Victoreen, Inc.
10101 Woodland Avenue
Cleveland, Ohio 44104
Next Calibration Due: April 18, 1985

Victoreen Traceability
Test Number DG8118/83
Calibration: September 29, 1983
PTW Chamber Model 30-343
Serial Number N23361-142

A-60-602

GEORGIA INSTITUTE OF TECHNOLOGY

CHARACTERIZATION OF CHANGES IN ADSORBENT PROPERTIES UNDER HIGH RADIATION DOSES

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CHARACTERIZATION OF CHANGES IN ADSORBENT PROPERTIES UNDER HIGH RADIATION DOSES

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October, 1985

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ABSTRACT

A research project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses," was undertaken by the Neely Nuclear Research Center, Georgia Institute of Technology, for the Savannah River Laboratory in June 1984 for about one year. The characterization consisted of measuring the following physical properties, as a function of dose, in silver and hydrogen zeolite: crystallinity, adsorption capacity, density, hardness, crushness, and possible chemical changes.

Although there were large deviations in the measurements, attributed to the poor quality of the mordenite, no adverse effects due to Co^{60} radiation doses of up to 1×10^{10} rads were found.

I. INTRODUCTION

A research project entitled "Characterization of Changes in Adsorbent Properties under High Radiation Doses," was undertaken by the Neely Nuclear Research Center, Georgia Institute of Technology, for the Savannah River Laboratory in June 1984 for one year. The characterization consisted of measuring physical changes in silver and hydrogen zeolite as a function of dose from Co^{60} radiation. Silver and hydrogen zeolites are molecular sieves which adsorb noble gases such as xenon and krypton. One possible application for these zeolites is containing and isolating radioactive xenon and krypton from the environment. In nuclear power plants radioactive xenon and krypton are produced continuously. Under normal operating conditions these gases are kept within the sealed tubes holding the fuel and thus are isolated from the environment. Under accident condition however, the radioactive gases may escape and if the flow of air within the containment building, which houses the reactor, were channeled through columns containing silver or hydrogen zeolite, the radioactive xenon and krypton gas would be held back and prevented from entering the environment. After all or most of the noble radioactive gases are retained in the zeolite, the column could then be valved off at both ends to bring about isolation from the environment.

Since the silver or hydrogen zeolite in the column would be in an intense radiation field originating from the decay of xenon and krypton, Savannah River Laboratory contracted with Georgia Tech to study the effects of high radiation doses on the physical properties of the zeolite. The physical properties which were measured as a function of dose were crystallinity, adsorption capacity, density, hardness, crushness, and possible chemical changes.

In this final report, the results of the year long study, which have been reported in monthly reports, are collected, analyzed and discussed.

II. SAMPLE HOLDERS AND EXPERIMENTAL CONDITION

Table I lists all sample holders, their numbers, content, and total dose exposure in rads. A total of 48 holders were made. Sixteen holders were control samples and 32 (16 silver and 16 hydrogen zeolite) were irradiated in batches of fours to 10^8, 1×10^9, 5×10^9, and 1×10^{10} rads. The first four control sample holders were designed to measure what happens to the air inside the stainless steel tubes which contained no zeolite but exposed to 1×10^{10} rads in one case and not exposed at all in the other. The next four control samples were used to monitor temperature and pressure on a daily basis for the entire exposure period. (There were no changes in the temperature or pressure as a function of dose. The data were simply constant.)

TABLE I. SAMPLE HOLDERS AND EXPERIMENTAL CONDITIONS

TYPE OF SAMPLE* HOLDER	SAMPLE HOLDER No.	QUANTITY	TOTAL DOSE RADS	EXPERIMENTAL CONDITIONS
Control	37	1	1×10^{10}	No Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Control	39	1	Not irradiated**	No Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Control	38	1	1×10^{10}	No Zeolite in sample holder, Saturated air (1001 PPM O_3; 1026 PPM NO_2)
Control	40	1	Not irradiated**	No Zeolite in sample holder, Saturated air (1001 PPM O_3; 1026 PPM NO_2)
Control	1 (gauged)	1	10^{10}	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Control	3 (gauged)	1	10^{10}	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Control	2 (gauged)	1	10^{10}	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Control	4 (gauged)	1	10^{10}	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	47 48	2	Not irradiated*	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	45 46	2	Not irradiated*	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	41 42	2	Not irradiated*	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	43 44	2	Not irradiated*	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	7 8	2	1×10^{8}	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	5 6	2	1×10^{8}	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	11 12	2	1×10^{8}	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	9 10	2	1×10^{8}	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	19 20	2	1×10^{9}	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	17 18	2	1×10^{9}	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	15 16	2	1×10^{9}	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	13 14	2	1×10^{9}	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)

TABLE I CONTINUED

Samples	23	24	2	5×10^9	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	21	22	2	5×10^9	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	27	28	2	5×10^9	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	25	26	2	5×10^9	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	35	36	2	1×10^{10}	Hydrogen Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	33	34	2	1×10^{10}	Hydrogen Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)
Samples	31	32	2	1×10^{10}	Silver Zeolite in sample holder, Sat. air (1001 PPM O_3; 1026 PPM NO_2)
Samples	29	30	2	1×10^{10}	Silver Zeolite in sample holder, Dry air (1001 PPM O_3; 923 PPM NO_2)

* Each sample holder consisted of one stainless steel valve (SSH4), 10 in. of 1/4" ss tube, and 1/2" swageloc ss-400-c end plug.

** Analyzed at the same time as irradiated samples

The next eight control samples were designed to measure what happens to the zeolite which is not irradiated but which is kept under the same conditions as the irradiated samples (i.e., under the same atmospheric conditions and in stainless steel tubes sealed and isolated from the environment. Two of the eight control samples were analyzed at the same time as each irradiated batch at each appropriate dose.

The initial gas composition in the dry sample holders came from a 10-liter glass container filled to 8.5 psig pressure containing 1001 ppm ozone and 923 ppm NO_2; the balance was dry air. The initial gas composition in the saturated sample holders came from another 10-liter glass container also filled to 8.5 psig pressure containing 1001 ppm O_3 and 1026 ppm NO_2; the balance was moisture saturated air. The glass containers were taped with black insulating tape to minimize interaction of NO_2 with light and to prevent shattering of the glass, in case of accident, since the container was pressurized to 8.5 psig.

Despite these efforts, Dr. Douglas Davis stated that the gas mixture forms an extremely active chemical system. The mixture is affected by light, temperature and surface conditions. The following reactions take place:

$$NO_2 + h \longrightarrow NO + O$$

$$O + O_2 \longrightarrow O_3$$

$$NO_2 + O_3 \longrightarrow NO_3 + O_2$$

$$NO_3 + NO_2 \longrightarrow N_2O_5$$

$$N_2O_5 + H_2O \longrightarrow 2HNO_3$$

$$NO_2 + H_2O \longrightarrow HNO_3$$

Reproducibility in systems containing these gases would be highly dependent on surface conditions when and if the light and temperature variables are carefully controlled. Dr. Davis believes controlling surface conditions is extremely difficult. Consequently, large fluctuation in the NO and NO_2 content of the gases in the sample holders was expected. Dr. Davis suggested that a useful index of what is happening to the gases in the tubes might be the sum of NO and NO_2 rather than the individual fractions of NO or NO_2. This index is used to reanalyze the data in an attempt to correlate the results.

The initial gas composition, as prepared in the glass containers by mixing nominally 1000 ppm NO_2 and 1000 ppm O_3 in dry and saturated air, was analyzed by Dr. Davis. His results were:

	NO	NO_2	O_3
Dry gas*, PPB**	3000	121	500
Saturated gas*, PPB	198	171	700

The uncertainty in the ppm's of NO, NO_2 and O_3 is 15%. The results of the initial gas analysis indicate that the presence of moisture appears to substantially affect the NO content. This fact cannot be explained by Dr. Davis' enumeration of the types of chemical reactions that take place.

III. CRUSHING TESTS

Tests designed to measure the crushing strength of objects are performed by placing the right cylindrical pellet of zeolite between two flat plates and the load is increased until the pellet is crushed. In these measurements it is necessary that a pellet has a smooth and even surface so that a line contact between pellet and flat plates is established. If the pellet surface is not smooth and not even, a point contact is established and the load needed to start the crushing process would be different from the case when a line contact exists. The quality of the surfaces of the pellets is then a variable that contributes to the spread in the crushing loads.

At first, ASTM standard D-4179-82 was chosen as the method of choice for measuring the crushing load for silver and hydrogen zeolite. This standard required, among other things, that samples be heated to 400 15 °C for not less than three hours. Initial measurement of the crushing loads on fifty pellets of each type of zeolite, conducted in accordance with the standard, showed very large deviations in the results. Table II shows the results. It was suspected that the heating and drying process was somehow making the pellets weak and contributing to the large variability in the crushing load. Consequently, another sample of fifty pellets of each type was used without being heated, to measure the crushing strength. The results are given in Table III. It is seen that heating and drying affects only silver zeolite making it weaker. As a result of this comparison, it was decided to forego the heating and drying step in the ASTM D-4179-82 standard for all subsequent analyses.

* Gas mixture content based on partial pressure makeup of standard dry air from Matheson and 923 ppm NO_2 and 1001 ppm O_3

* Gas mixture content based on partial pressure makeup of standard air from Matheson that was saturated with H_2O + 1026 ppm NO and 1001 ppm O_3.

TABLE II. ZEOLITE CRUSHING LOADS (kgf)*
(Heated and Dried)

Hydrogen Zeolite		Silver Zeolite	
5.36	8.86	5.81	1.57
3.03	12.36	1.60	0.70
7.02	2.62	0.92	0.75
5.95	4.47	5.34	0.69
3.05	3.22	0.51	5.72
4.08	6.50	2.00	6.43
5.08	2.78	4.83	6.50
1.98	2.30	0.59	1.85
10.28	9.93	0.18	1.14
5.63	2.89	2.91	8.25
5.83	17.04	0.20	9.57
4.75	13.50	2.18	3.90
6.00	3.63	0.94	0.99
3.49	5.60	2.99	4.71
7.14	5.61	0.80	6.86
2.84	2.74	5.60	0.50
2.70	11.84	0.62	0.20
6.81	5.21	2.21	1.58
9.39	5.60	0.19	1.71
2.48	7.36	3.52	1.00
1.86	8.79	2.18	2.36
7.44	9.41	17.14	1.37
5.64	6.80	0.59	0.96
8.59	2.04	10.44	2.14
4.18	5.97	0.43	1.58
$\bar{x}$	5.953		2.955
S	3.285		3.289
80% Spread	1.748 - 10.158		0.0 - 7.165
95% Reliability	5.042 - 6.864		2.043 - 3.867

*Comparison of these crushing strengths using a two tailed t-test indicates that there is a highly significant difference in their crushing strengths. The samples tested were between 0.23 and 0.52 inches in length. There was not a significant correlation between length and crushing load. Tests were carried out according to ASTM D 4179. The applied rate of force was increased at 4.5 lbf/sec.

TABLE III. ZEOLITE CRUSHING LOADS (kgf)
(Undried and Unheated)

Hydrogen Zeolite		Silver Zeolite	
4.47	8.82	4.90	2.25
5.26	2.17	6.06	17.62
2.83	2.78	5.30	10.76
5.20	4.24	5.13	1.65
7.17	2.59	8.00	2.30
2.33	4.15	5.97	5.89
4.62	2.60	14.89	9.12
9.72	4.79	4.81	1.66
5.68	5.41	4.02	4.32
8.60	1.97	0.52	4.50
3.36	3.55	3.53	2.41
8.01	2.44	1.20	0.62
4.95	5.51	1.71	0.90
4.63	5.04	6.41	2.84
7.96	1.49	2.20	0.83
6.26	2.27	1.94	12.36
8.84	1.75	2.84	11.64
5.20	5.52	3.53	10.04
8.46	2.52	4.63	4.87
6.08	3.72	6.94	0.71
2.25	9.77	2.51	1.72
6.87	8.93	2.63	1.60
8.84	4.88	12.52	1.01
3.90	2.57	2.11	1.31
3.22	2.94	8.52	0.53

$\bar{x}$	4.943	4.726
s	2.384	4.040
80% Spread	1.891 - 7.994	0.0 - 9.897
95% Reliability	4.282 - 5.604	3.606 - 5.846

Comparison of these crushing strengths and those of the dried samples using a two tailed t-test indicates that the only significant difference was between the dried and undried silver zeolite samples. The samples tested were between approximately 0.25 and 0.50 inches in length. These tests were carried out according to ASTM D 4179 except the samples were not dried. The applied rate of force was increased at 4.5 lbf/sec.

IV. HARDNESS TESTS

Tests designed to measure the load needed to penetrate the pellet surface at a point are known as hardness tests. The apparatus used is a diamond pyramid indenter which presses on the pellet at a point rather than at a line as in the case of the crushing tests. The purpose of this test is that if the packing in a column is such that contacts among pellets occur at points, rather than lines, then it is useful to know what loads are needed to affect penetration.

The hardness tests were performed using a Tukon Tester manufactured by Wilson Mechanical Instrument Division of the American Chain and Cable Company. Before measuring each zeolite pellet's Knoop Hardness Number, defined as a unitless quantity relating the load in kgf per length^2 of indentation, the bottom of the pellet was flattened on 189 grit SiC sandpaper and the top was ground lightly on 600 grit SiC. Typical hardness results for initial unirradiated pellets are given in Table IV.

V. DENSITY MEASUREMENTS

Pellets were broken into small pieces approximately 1/4" long. Batches about two grams of each zeolite were dried at 400° F for four hours and then cooled in a desicator. After cooling, each batch was weighed to the nearest 0.0001 gram. The batch was then soaked in kerosene for 24 hours. This procedure allows the kerosene to penetrate all the pores in the zeolite. The weight of the batch suspended in kerosine is then determined to the nearest 0.0001 gram. The density of the zeolite batch is obtained as follows:

$$\text{Volume of Batch} = \frac{\text{Weight in air - weight surrounded in kerosine}}{\text{density of kerosine}}$$

$$\text{Density of Batch} = \frac{\text{Weight in Air}}{\text{Volume of Batch}}$$

Triplicate determinations were made on each sample of zeolite. Table V lists the initial densities of the fresh zeolite.

Table V. Density Measurement of Fresh Hydrogen and Silver Zeolite

Hydrogen	Silver
1.8548	2.3255
1.8601	2.3259
1.8449	2.3243
Average = 1.8549	Average = 2.3252

TABLE IV. MICROHARDNESS (KNOOP HARDNESS NUMBER)*

Hydrogen		Silver	
92.0	89.8	74.1	22.7
53.5	143.8	52.1	21.9
95.3	91.0	58.8	19.5
106.8	63.3	89.0	28.1
57.2	86.0	12.4	20.6
73.8	78.1	17.3	14.4
135.0	80.2	61.8	31.3
194.1	127.0	42.5	24.1
102.9	110.8	32.7	65.2
235.1	93.6	42.5	29.2
$\bar{x}$ = 105.5		$\bar{x}$ = 38.0	
S = 44.7		S = 21.9	

*These tests were done on a Tukon Tester manufactured by Wilson Mechanical Instrument Division of American Chain and Cable Company using a 175 gram load. The bottom of the samples were flattened on 180 grit SiC paper so they were stable on the testing stage. The tops of the samples were very lightly ground on 600 grit SiC paper before the hardness measurements were made.

VI. ADSORPTION MEASUREMENTS AND X-RAY DIFFRACTION CALIBRATION

The adsorption measurements were carried out using the McBain-Baker gravimetric system which employs a spiral quartz spring on which a sample is suspended in a pan. The extension of this spring is a direct measurement of the weight of the adsorbed gas. These springs were periodically calibrated with standard weights.

Oxygen adsorption in the fresh hydrogen and silver zeolite was measured at 75 torr and -196 °C. The results were:

H-zeolite (grams of oxygen adsorbed per 100 grams of zeolite) = 22.7
Silver-zeolite (grams of oxygen adsorbed per 100 grams of zeolite) = 9.2
Standard H-zeolite = 22.6

The X-ray diffraction equipment was calibrated daily with an internal standard. Diffraction patterns of two, fresh H- and Ag-zeolite samples were obtained and compared to a hydrogen zeolite standard with 100% crystallinity. The results were:

H-zeolite crystallinity = 79.0%
Ag-zeolite crystallinity = 16.0%

The X-ray pattern of the Ag-zeolite had several unidentified peaks.

VII. QUALITY OF ZEOLITE AS RECEIVED FROM SRL

Dr. Tudor Thomas, a recognized expert in zeolite, has stated that the zeolite which SRL supplied Georgia Tech with was produced from sodium mordenite pellets, which were obtained by mixing sodium mordenite with 20% clay and extruding. The sodium atoms in the sodium mordenite pellets were then exchanged with either hydrogen and silver zeolite respectively. This procedure, according to Thomas, often results in an incomplete exchange of sodium for either hydrogen or silver atoms. Consequently, the quality of the zeolite in general and of silver zeolite in particular left a lot to be desired. Dr. Thomas supports his observation by the fact that when pellets of either hydrogen or silver zeolite are broken, cores of various sizes in the center of pellets are seen. The variation in the size of cores is attributed to non-uniform exchange. The presence of the core indicates an incomplete exchange.

The non-uniform makeup of the zeolite is thought to cause the large variation in crushing strength, hardness, and surface conditions.

Dr. Thomas recommends that the mixing with clay and pelletizing by extrusion should be done after all the sodium atoms have been replaced completely with either hydrogen or silver in the powder form.

VIII. RESULTS AND CONCLUSIONS

Table VI summarizes all the results. Average values of two or more samples are quoted. The original sample numbers quoted in Table I and reported in monthly reports appear in parentheses in the left-most column. Some observations follow:

- The NO and NO_2 content of sample holders 39 and 40 (i.e., empty tubes [no zeolite] with gas only) with no exposure to Co^{60} radiations was higher than the content of all the other sample holders which contained zeolite. This indicated that either a chemical reaction or adsorption by zeolite was responsible for the depletion of NO and NO_2.

- The exposure of sample holders 37 and 38 to 1×10^{10} rads with again only dry and saturated gas left the highest concentration of NO and NO_2 in the tubes (193,000 ppb for dry gas and 135,000 ppb for saturated gas, see Table VI). This indicated that the radiation field affected the quality of the gas in the empty tubes without zeolite and that moisture depletes the NO and NO_2 content somewhat.

- No correlation is apparent between NO and NO_2 content in zeolite filled tubes on one hand and exposure dose on the other. The variation in the data is large. This is also true for dry and saturated gas. The cause of the variation in the data may be due to the poor quality of the zeolite.

- There appears to be an increase in the density of the zeolite which was placed in sample holders. The increase is not related to exposure dose. The initial density of the fresh hydrogen zeolite was 1.855 g/cm^3 and of fresh silver zeolite was 2.325 g/cm^3. The range of densities for hydrogen zeolite, as tabulated in Table VI, is 1.890 - 2.007 g/cm^3. Similarly, the range of densities for silver zeolite is 2.340 - 2.444/cm^3.

- There may also be a slight increase in crushing strength and hardness in silver zeolite and to a lesser degree in hydrogen zeolite which cannot be correlated with exposure dose. The minimum and maximum values of crushing load in Table VI for hydrogen- and silver-zeolite are 5.9-9.4 and 7.1-12.3 kgf respectively. The initial average crushing load for fresh hydrogen zeolite was 4.94 kgf with minimum and maximum values of 1.97 and 9.77 kgf. For

silver zeolite the average value was 4.73 with minimum and maximum values of Ø.53 and 14.89 kgf. Again the large variation in data makes it difficult to draw firm conclusions. Similar trends are observed in the hardness data.

- The percent crystallinity was reasonably consistent with hydrogen zeolite having a range of 7Ø-84% and silver zeolite having a range of 15-19%. The exposure dose did not seem to affect crystallinity.

- The adsorption capacity was also consistent with hydrogen zeolite having a range of 21.9-25.2% and silver zeolite having a range of 8.1-13.8%. Again, exposure dose did not appear to affect adsorption capacity.

TABLE VI SUMMARY OF ALL RESULTS

Samples (Original Sample #)	GAS ANALYSIS NO(PPB)	NO_2(PPB)	NO + NO_2(PPB)	DENSITY Density (g/cm^3)	CRUSHING LOAD kgf	HARDNESS (Knoop Hardness No.)	CRYSTALLINITY %	OXYGEN ADSORPTION %
	DOSE = 1.0×10^{10} Rads			DOSE = 1×10^{10} Rads	DOSE = 1×10^{10} Rads	DOSE = 1×10^{10} Rads	DOSE = 1×10^{10} Rads	
Dry Ag Zeolite (1,29,30)	43	240	283	2.417	10.6 ± 7.5	40.9 ± 7.8	19.3	13.8
Sat. Ag Zeolite (2,31,32)	48	397	445	2.375	12.3 ± 6.7	46.6 ±19.2	19.3	12.8
Dry H Zeolite (3,33,34)	3020	1083	4103	1.912	9.4 ± 5.3	90.9 ±34.6	79.3	25.2
Sat. H Zeolite (4,35,36)	6469	3220	9688	1.932	9.2 ± 2.9	117.0 ±35.0	84.5	23.6
Dry Gas Only (37)	106000	87000	193000					
Sat. Gas Only (38)	72000	63000	135000					
CONTROL SAMPLES	DOSE = 0.0			DOSE = 0.0	DOSE = 0.0		DOSE = 0.0	DOSE = 0.0
Dry Gas Only (39)	10100	24000	34100					
Sat. Gas Only (40)	3500	9100	12600					
Dry Ag Zeolite (42)	501	2500	3001	2.391*	8.2 ± 2.3	50.0 ±33.5	20.6	12.0
Sat. Ag Zeolite (44)	280	1700	1980	2.404	10.4 ± 6.1	35.4 ± 7.1	21.3	12.7
	DOSE = 5×10^9 Rads			DOSE = 5×10^9 Rads	DOSE = 5×10^9 Rads		DOSE = 5×10^9	DOSE = 5×10^9
Dry Ag Zeolite (21,22)	26	145	171	2.350	9.5 ± 3.7	40.6 ± 8.1	17.5	9.7
Sat. Ag Zeolite (23,24)	15	45	60	2.340	10.3 ± 5.1	39.9 ±14.3	18.0	8.1
Dry H Zeolite (25,26)	25	510	535	1.890	9.2 ± 1.6	103.1 ±22.5	73.0	22.7
Sat. H Zeolite (27,28)	24	51	75	1.906	9.2 ± 3.6	75.9 ±25.6	78.0	23.6
CONTROL SAMPLES (Dose = 0.0)				DOSE = 0.0	DOSE = 0.0		DOSE = 0.0	DOSE = 0.0
Dry H Zeolite (46)	9	180	188	1.940	7.2 ± 2.1	113.0 ±31.6	84.0	23.2
Sat. H Zeolite (48)	9	40	49	1.918	11.4 ± 4.6	95.9 ± 9.3	70.0	26.2
	DOSE = 1×10^9 Rads)			DOSE = 1×10^9 Rads	DOSE = 1×10^9		DOSE = 1×10^9	DOSE = 1×10^9
Dry Ag Zeolite (13,14)	13	375	388	2.435	11.0 ± 3.4	38.6 ±10.8	16.8	13.3
Sat. Ag Zeolite (15,16)	9	215	224	2.440	10.5 ± 3.8	40.1 ±11.3	16.6	14.2
Dry H Zeolite (17,18)	9	265	274	1.976	9.9 ± 4.6	124.5 ±68.0	71.8	23.5
Sat. H Zeolite (19,20)	27	225	252	1.985	8.3 ± 2.8	109.7 ±22.8	78.7	23.2
CONTROL SAMPLES (Dose = 0.0)				DOSE = 0.0	DOSE = 0.0	DOSE = 0.0	DOSE = 0.0	DOSE = 0.0
Dry H Zeolite (45)	31	90	121	1.971	5.9 ± 2.6	110.9 ±24.1	69.8	22.4
Sat. H Zeolite (47)	46	50	96	1.963	10.1 ± 3.1	143.7 ±47.1	74.5	23.6
	DOSE = 1×10^8 Rads			DOSE = 1×10^8 Rads)	DOSE = 1×10^8 Rads	DOSE = 1×10^8 Rads	DOSE = 1×10^8	DOSE = 1×10^8
Dry Ag Zeolite (5,6)	83	511	594	2.444	7.9 ± 4.8	41.9 ±14.2	17.5	9.3
Sat. Ag Zeolite (7,8)	20	168	188	2.437	7.1 ± 3.8	40.4 ± 7.3	17.0	10.1
Dry H Zeolite (10)	37	491	528	2.007	6.2 ± 2.3	100.1 ±18.6	87.0	22.4
Sat. H Zeolite (11,12)	20	250	270	1.974	6.2 ± 2.4	74.1 ±23.0	79.5	21.9
CONTROL SAMPLES (Dose = 0.0)				DOSE = 0.0	DOSE = 0.0		DOSE = 0.0	DOSE = 0.0
Dry Ag Zeolite (41)	21	216	237	2.443	9.9 ± 2.4	43.9 ± 4.4	16.0	10.8
Sat. Ag Zeolite (43)	26	122	148	2.436	9.9 ± 3.2	47.5 ± 6.2	15.0	12.3